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AFFDL-TR-77-51



## DEVELOPMENT OF CAST CARCASS TIRES FOR MILITARY AIRCRAFT

ZEDRON, INC. 4211 MENLO DRIVE BALTIMORE, MARYLAND 21215

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**JULY 1977** 

TECHNICAL REPORT AFFDL-TR-77-51 Interim Report for Period September 1976 — June 1977



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This technical report has been reviewed and is approved for publication.

PAUL C HERICH

Project Engineer

Vehicle Equipment Division

Air Force Flight Dynamics Laboratory

HOWELL K. BREWER, Chief

Lowell K. Khew

Mechanical Branch

Vehicle Equipment Division

Air Force Flight Dynamics Laboratory

FOR THE COMMANDER

AMBROSE B. NUTT, Director

Vehicle Equipment Division

Air Force Flight Dynamics Laboratory

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## 20. ABSTRACT (Cont'd.)

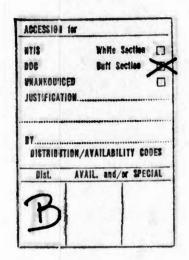
Utilizing the results obtained from the materials study, a plan for Phase II accomplishment was outlined and an initial iteration design and construction was selected for the one piece cast tire and cast carcass/replaceable tread tire.

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#### FOREWORD

This report was prepared by R. K. H. Eggers, E. J. Georgia and E. L. Molnar of Zedron, Inc. and G. C. Derringer, M. M. Epstein and W. J. Mueller of Battelle Columbus Laboratories, under USAF Contract F33615-76-C-3062. The work was conducted under the direction of the Vehicle Equipment Division, Air Force Flight Dynamics Laboratory, Wright-Patterson Air Force Base, Ohio, and George J. Sperry and Paul C. Ulrich (AFFDL/FEM), Project Engineers. This report covers work performed in Phase I between September 1976 - June 1977. The report was submitted by the author July 1977 for publication as a technical report.





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## DEVELOPMENT OF CAST CARCASS TIRES FOR MILITARY AIRCRAFT

## INTERIM REPORT - PHASE I

## SECTION I

## INTRODUCTION

This report describes work undertaken by Zedron, Inc. during Phase I of a three-phase program to demonstrate the feasibility of using a one piece cast tire and/or a cast carcass replaceable tread tire on military aircraft. Work was done during the period September, 1976 through June, 1977. The Phase I scope, as defined in Contract No. F33615-76-C-3062, consists of "materials survey and evaluation, the preparation of preliminary design drawings and specifications, and the preparation of a plan for Phase II accomplishment." The primary work on the materials survey and evaluation portion of Phase I was accomplished by the Battelle Columbus Laboratories (BCL) under a Zedron subcontract.

Several advantages have long been recognized for a cast military aircraft tire compared with conventional tire constructions. The large amount of hand lay-up work in conventional tire manufacture is not only costly, but the quality of the resulting tire varies with the skill, or lack of skill, of the individual tire builder. The cast tire should not only reduce the original cost, but also reduce the

possibilities for human error. Conventional aircraft tires, wear tread very rapidly and are retreaded several times. This represents a high logistic and labor cost. It is expected that a replaceable tread on the cast carcass can be changed without removing the wheel from the aircraft, thus reducing maintenance time. The present system also presents serious logistic problems in the storage of many tire sizes in many locations and the need to have equipment and trained personnel at all locations to make tire changes. Replaceable treads should greatly reduce this problem.

TERROREA VALUE IEN HOT BETTE BRACKED TRES TO TERROROGISTAN

The concept of cast tires has been investigated to some extent by the Air Force Flight Dynamics Laboratory with the earlier results reviewed in two reports. These are AFFDL-TM-74-154-FEM, "Analysis of Thermoplastic Elastomer Tire", James R. Hampton, August, 1974, and AFFDL-TM-74-198-FEM, "Test Results of a Castable Tire", James R. Hampton, October, 1974. While the results of the work did not meet all expectations, they were encouraging enough to continue additional investigations. The current program is an outgrowth of these earlier studies.

The program as outlined was concerned with two basic tire configurations. These are (1) a one piece cast tire and (2) a cast carcass with a replaceable reinforced rubber tread. Because the basic carcass was expected to be similar in each case, the work on the two types of tires was carried on simultaneously for greater efficiency.

The long-term goal of the three-phase program is to demonstrate that a cast carcass tire is a practical concept for military aircraft. As such, work on materials for the application is not expected to result in an "ideal" system, but is structured instead toward the goal of identifying practical material concepts. It would be somewhat surprising if a material ideally suited for the application were to be immediately available from commercial sources. Hopefully, the research and development effort will not only confirm the validity of the concept, but will also provide insights into the characteristics of preferred polymers and compounds that will be capable of providing superior field performance.

The closed torous, one component cast tire design is unique unto itself in its shape, construction and operating mode. Special tire design parameters need to be intimately integrated with material properties and since conventional tire design practice is not applicable, new design and operating standards need to be more fully explored and evaluated.

## SECTION II

#### SUMMARY

The primary objective of the Phase I program was to survey and evaluate materials for potential use in cast tires for military aircraft. This was accomplished by selecting candidate polymers, and evaluating these polymers, with and

without reinforcing fillers. The evaluation was based on properties which demonstrate the ability to perform in the cast tire application. Many contacts were made with polymer and filler vendors to obtain their input in selecting materials for evaluation.

The initial screening of polymer-filler systems was directed to identifying combinations which had sufficient stiffness (as measured by Young's Modulus) and with sufficient elongation to be functional. To achieve this, it was found that the system required a hardness of approximately 65 Shore D. This was attained in the hardest polyester, 55D polyesters with a glass filler, and some of the very hard polyurethanes containing a glass filler.

Polymer-filler combinations were evaluated by other criteria. Three systems have been selected to initiate the tire preparation program in Phase II. These are described in Table 1. Two are based on a thermoplastic copolyester elastomer and the third on a thermosetting polyurethane.

One of the copolyester elastomers and the polyurethane are reinforced with milled glass, while the second copolyester is a harder material which does not require a filler.

Based on the results obtained from the materials study, a plan for Phase II accomplishment was outlined and an initial iteration design and construction was selected for the one piece cast tire and cast carcass/replaceable tread tire.

The first prototype one piece cast tire will be a relatively low profile design with an aspect ratio of 0.65 and cast from an unmodified copolyester elastomer with an approximate hardness of 65 Shore D.

The first prototype cast carcass for the two piece replaceable tread tire will also be a relatively low profile design and cast from an unmodified copolyester elastomer with an approximate hardness of 55 Shore D. The replaceable tread will be formulated from a natural rubber compound reinforced with continuous aramid cords.

## SECTION III

## METHOD OF APPROACH

To attain the objectives of the Phase 1 program (surveying, evaluating, and choosing candidate materials for producing cast tires for military aircraft) the following approach was developed and used throughout the program:

- (1) Outline criteria for evaluation of materials
- (2) Contact vendors to obtain polymer and reinforcements for study in the program.
- (3) Select polymers and reinforcements for evaluation.
- (4) Determine basic properties of candidate polymers to determine suitability for application.
  - (5) Study the effect of reinforcements and modifiers in candidate polymers.

- (6) Study effect of processing techniques on material properties.
- (7) Choose three candidate compositions as starting materials for the preparation of test tires in Phase II.

## 1. Evaluation Criteria

One of the first tasks in the program was to specify the criteria by which the candidate polymer systems could be judged. Since there are no specifications available for materials for these constructions, much emphasis had to be placed on limited information available from earlier tire tests at AFFDL, guidance from Zedron based on its past experience, and background experience from the earlier tire component developments programs at BCL. Experience at Zedron had suggested that the carcass material ought to be about 50 percent stiffer than the polyester used in earlier tests of the cast carcass concept. This material had a reported Young's modulus of about 40,000 psi. It was recognized that an increase in stiffness would probably be at the sacrifice of other properties including yield elongation, and that it would be necessary to obtain the best balance among several properties. It was also known that earlier cast tires had failed during dynamometer tests. Limited information available regarding these failures suggested that a fatigue or dynamic fatigue mechanism was involved.

In addition to the static mechanical properties, it was recognized that many other characteristics are very important in the evaluation of materials for potential applicability to cast tire constructions, although exact numbers could not be assigned to these parameters. Key properties chosen for study and the rational for these choices follows.

Tear Strength - a cast tire with no reinforcement other then the polymer-filler system must have high tear strength to resist the high internal and external stresses.

Flex crack resistance - repeated rapid flexing during landing and takeoff operations has the effect of initiating and propagating cracks, primarily in the tire sidewall. This measurement was made to determine crack resistance to this type of failure.

Coefficient of friction - this property is of importance in the one piece cast tire and the coefficient must be sufficient to maintain control of the aircraft during movement on the ground under varied conditions.

Abrasion resistance - this property is of importance for the one piece cast tire since the tire must have a reasonable useful life.

Resilience - (dynamic mechanical behavior) - aircraft tires are subject to many deformations during landing and takeoff operations, and this property provides an indication of the ability of a material to absorb shock loadings.

Hysteresis - flexing which takes place during landing and takeoff operations generates heat due to hysteresis in the tire materials. This heat can shorten tire life or even cause catastrophic failure if excessive.

RECOMMENDED POLYMER SYSTEMS FOR INTITAL PHASE 2 TIRE PREPARATION TABLE 1.

24-5 Polymer B filled with S600 1700 7 2680 20 2400 86 50.00  46-4 Polymer I filled with 2700 2400 8 3250 40 3200 50 57,100  47-1 Polymer I filled with 2700 2300 8 3250 40 3200 50 57,100  47-1 Polymer I filled with 2700 2300 8 3250 40 3200 50 67 66,700  24-5 Polymer I filled with 2700 2300 8 3250 40 3200 50 67 66,700  24-5 Polymer I filled with 64 590 50 724 117 1.07 49 (set) 59.7 x 10 <sup>5</sup> 51.3 x 10 <sup>5</sup> (s.)  24-6 Polymer I filled with 64 590 50 724 117 1.07 49 (set) 59.7 x 10 <sup>5</sup> 51.3 x 10 <sup>5</sup> (s.)  24-7 Polymer I filled with 64 590 50 724 1.00 40.8 8 50.7 x 10 <sup>5</sup> 51.3 x 10 <sup>5</sup> (s.)  24-7 Polymer I filled with 64 590 50 70 724 1.17 1.07 49 (set) 59.7 x 10 <sup>5</sup> 51.3 x 10 <sup>5</sup> (s.)  24-8 Polymer I filled with 64 590 50 70 724 1.17 1.07 49 (set) 59.7 x 10 <sup>5</sup> 51.3 x 10 <sup>5</sup> (s.)  24-9 Polymer I filled with 75 68 1030 33 28 989 1.00 98 1.00 98 x 10 <sup>5</sup> 51.3 x 10 <sup>5</sup> (s.)  24-1 Polymer I filled with 55 630 13 35	Composition	Identification	Stress at 10%   Elongation, ps1	Proportional Limit, pst	Elongation at Proportional Limit, 7	at Yield al Strength, psi	Elongation at Yield,	ion Tensile ld, at Break, psi	le Elongation ak, at Break,	ak, Hardness,	Young's Modulus, psi	
Folymer C	24-5	Polymer B filled with 20 phr milled glass fiber (Pl178-1/4)	2600	1700	2	2680	20	2400		29	66,700	
Polyment   Figure	7-97	Polymer C	2700	2400	80	3400	25	4700		63	57,100	
Compression   Tear   Strongth, Rebound, Buildup   Abrasion of Friction   Tear   Cracking Resistance   Compression   Strongth, Rebound, Buildup   Abrasion of Friction   Transition Of Friction Of Friction   Transition Of Friction Of Fr	1-27	Polymer I filled with 20 phr milled glass fiber (Pl178-1/4)		2300	80	3250	07	3200		29	66,700	
24-5 Polymer B filled with 64 590 50 20 724 1.17 1.07 -49 (est) 59.7 x 10 <sup>5</sup> 51.3 x 10 <sup>6</sup> 20 phr miled glass fiber (PlI7B-1/4)   46-4 Polymer I filled with 55 630 13 35	Composition	Identification	Compression Set, 25 percent deflection, 7	S	1 1			Coefficient of Friction Dry Wet		10	Crack Crack on	istance Cycles to Failure
46-4 Folymer C 68 1030 33 28 989 1.84 0.82 8 9.8 x 10 <sup>5</sup> / <sub>3</sub> (4) <1.8 x 10 <sup>5</sup> / <sub>4</sub> (4) <1.8 x 10 <sup>5</sup> / <sub>4</sub> (4) <1.8 x 10 <sup>5</sup> / <sub>4</sub> (1) <1.8 x 10 <sup>5</sup> / <sub>5</sub> (1) x 10 <sup></sup>	24-5	Polymer B filled with 20 phr milled glass fiber (Pll7B-1/4)	79	290	20		42					.3 × 10 <sup>6</sup>
47-1 Polymer I filled with 55 630 13 35	7-97	Polymer C	89	1030	33		68	*			© (3)	.8 × 10 <sup>6</sup> .3 × 10 <sup>6</sup>
Duplicate samples.	47-1	Polymer I filled with 20 phr milled glass fiber (Pl178-1/4)	\$5	630	13	35	ona i	en ett	de 18		am dad	E 02
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Compression set - aircraft tires are often idle for long periods under the combined loading of tire pressure and aircraft weight. If the tire material has high set, this will result in a permanent deformation. Similarly, stress relaxation measurements also provide an indication of the dimensional stability of the material under load. These time-dependent properties can also be used to determine the effectiveness of various reinforcing concepts.

Stress cycling - this test measures hysteresis and permanent set as well as their change upon repeated cycling. It is important because stress strain behavior of polymeric materials and especially filled polymers can show a large dependence upon previous stress strain history. The change in permanent set with repeated cycling is a rough measure of dynamic creep and is important because dynamic creep is a possible failure mode for a cast tire.

Glass transition temperature - the amorphous portion of a polymer changes from elastomeric to glassy as the temperature is decreased through the glass transition temperature. This property then gives an indication of the effects which can be expected when the article in question functions at a reduced temperature.

In addition to the above, the materials of construction must also satisfy minimum requirement for chemical and weathering resistance. The latter characteristic was ignored, because it is a property that can be improved through proper compounding, and this program is not structured to develop optimum compositions. Thus, long-term effects such as damage caused by heat (solar radiation) and sunlight were not considered in the selection process. However, rapid effects, such as crazing due to exposure by hydrocarbon fluids influenced the selection process, although no measurements of this property

were attempted.

#### 2. Material Selection

After the evaluation criteria had been outlined, a major effort was made to contact polymer and filler vendors to obtain recommendations for select materials to be used in this program, and to obtain relevant property and processing data.

Three basic types of polymers were eventually studied in this investigation, as outlined in Table 2.\* These were thermoplastic polyester, polyurethanes, and low molecular weight cross-linkable polybutadiene. A fourth class of material, the block copolymers of styrene and butadiene, was considered but was dropped because the materials failed to meet some of the basic criteria for processing, mechanical properties, or chemical resistance. Polyesters were included because of favorable experience with this kind of material in earlier work by Zedron and in wheel tests conducted by AFFDL. Polyurethanes (both thermosetting and thermoplastic) cover a very wide range of properties. Some of these materials are very tough and abrasion resistant and could be expected to meet many of the criteria set for the cast tire material.

<sup>\*</sup> Specific polymers studied in this program are not identified by trade name and are coded with letters A-L (see Qualifications).

TABLE 2. POLYMERS AND FILLERS INVESTIGATED

Code No.	Polymer
A	40D polyester
В	55D polyester
С	63D polyester (supplied by Zedron)
D	72D polyester
E	55D polyester (development material)
F	55D polyester (supplied by Zedron)
G	polyether-based TDI terminated urethane prepolymer
H	MDI-based polyurethane
I	MDI-based urethane prepolymer
J	polyether-based TDI terminated urethane prepolymer
K	thermoplastic polyurethane
L	crosslinkable polybutadiene
	Filler
1	419AA - 3/16-inch chopped glass fiber (OCF)
2	P117B - 1/4-inch milled glass fiber (OCF)
3	P117B - 1/8-inch milled glass fiber (OCF)
4	P117B - 1/16-inch milled glass fiber (OCF)
5	3531 - 1/8-inch chopped glass fiber (PPG)
6	B-30-B glass bubbles (supplied by Zedron)
7	1/16-inch glass beads (supplied by Zedron)
8	1/32-inch glass beads ( ditto )
9	1/64-inch glass beads ( " )
10	Hi-Sil 233 (PPG)
11	Cab-O-Sil M5 (Cabot Corporation)
12	Calcene TM (PPG)
13	Calidria HPP asbestos (Harwick)
14	Santoweb H (Monsanto)
15	Santoweb W (Monsanto)
16	N-330 carbon black (several suppliers)
17	micronized talc (W. H. Loomis Company)
18	aluminum flake (BCL preparation)

Polybutadiene was considered because of extensive evaluation in cast automotive tires, and the variety of types currently available with high chemical reactivity to produce a range of properties. Very little work was done with polybutadiene, however, as the initial results with the available materials showed that they fell far short of the objectives of this program.

As the most viable candidate polymers were identified, consideration was given to the upgrading of properties through the addition of reinforcing fibers or fillers. These included materials with a high aspect ratio such as glass fiber, cellulosic fiber, and asbestos fiber. Lower aspect ratio fillers such as silica, fine calcium carbonate, glass bubbles, and carbon black were also studied. The glass fiber was considered a prime candidate filler and was examined extensively in several forms, including chopped strand and milled strand. All modifiers studied in this program are described in Table 2.

Considerable effort was also directed to the method of incorporating the fillers in the polymers, particularly the polyester. Dry powder blending (for solids) as well as hot milling techniques were examined.

Phase I culminated with the selection of three compositions for tire prototype testing in Phase II. These are not to be considered optimum compositions, but legitimate

starting points for evaluation of promising compositions.

Data from the Phase II testing will undoubtedly reveal shortcomings of these prototype cast tire systems, perhaps both in composition and design. An iterative process, based on an improved understanding of the requirements for this novel concept, would then begin to upgrade performance both through changes in materials and tire design.

It should also be noted that the exploratory investigations described in this report did not always result in desirable properties. Some surprises were encountered, as will be noted in later sections of the report, and there is little doubt that many properties could be improved by additional research with materials (polymers and reinforcement) specifically tailored to the application. That is to say, working with commercially available materials imposed serious constraints on the technical progress toward achieving the long-term goal of AFFDL. These constraints, and some possible solutions, are discussed in more depth elsewhere in this report.

## SECTION IV

## RECOMMENDATIONS

Three compositions from the Phase 1 program are recommended for initiating the Phase II tire fabrication program. Two are based on a thermoplastic polyester (one unmodified polymer and one modified polymer) while the third

is based on a modified thermosetting polyurethane. In each case, the modification consists of the incorporation of milled glass fiber. Details of the compositions are given in Table 1.

## SECTION V

## CONCLUSIONS

Studies undertaken in the Phase 1 program to evaluate for potential use in cast tires for military aircraft have led to the following conclusions.

- (1) Based on criteria specified for this program, the selected thermoplastic polyester and thermoset polyurethane materials have the potential for use in the carcass of the cast tire.
- (2) Frictional characteristics may restrict the use of the polyester and polyurethane materials to the carcass with a replaceable tread in an alternate material.
- (3) Generally, reinforcing fillers are required to develop the desired level of stiffness in the carcass material.
- (4) Glass fibers, particularly milled, are the most effective fillers evaluated and have the best potential for use in the rotational casting system.
- (5) Compositions have been prepared either by hot milling or powder blending which have the same degree of reinforcement. The powder blending, of course, is much more adaptable to the rotational casting process.

As noted in the introduction, it was not anticipated that this exploratory experimental study would result in an ideal system for the application. Until tire testing in Phase II of the first prototypes is completed, any deficiencies of the recommended systems cannot be ascertained. However, several comments regarding the recommended materials, and probable areas for further study are in order.

- Since the program was limited to commercially available materials, or those in an advanced state of development, consideration should be given to the eventual development of polymers specifically structured for this application. This should not be undertaken until at least the Phase II results are known and more data are available on tire performance with the three systems recommended by this program. The Phase II data will help delineate more clearly the specific properties required for the application.
- The polyester elastomers are very interesting materials for this application. The 55 D durometer material failed prematurely in dynamometer testing several years ago, and a 63 D durometer material is among those recommended for Phase II testing. The composition of these polyesters are not specifically known, and has not been totally disclosed by the manufacturer. However, they are believed to be copolymers, consisting of a hard phase (plastic) and a soft, dispersed phase (elastomer). The hard phase is dominant, as evidenced by the stress strain characteristics of the polymers. It is noted that the 63 D durometer material has a relatively high  $T_q$ , which may prove to be detrimental. Thus, á composition intermediate between the 55 D and 63 D polymers could be better suited for the application.

- Assuming the polyesters continue to show promise for cast carcass tire applications, the Phase 1 results suggest that more research will be needed on the problem of reinforcement. The data suggest that silane treatment of glass, as embodied by both commercially available reinforcements and the silane used to treat the glass bubbles, may not be the best approach to maximizing interaction with the polymer matrix. Research on this subject might also lead to methods of effective reinforcing with carbon black and other particulate fillers. Also, if these materials are chemically joined, phase dispersed polymers, as suggested by the previous paragraph, should be undertaken to determine the specificity of reinforcements for each phase. The experimental data suggest that the main effect obtained in this study is reinforcement of the plastic or hard phase, but the soft phase is unaffected, and modification of the latter may be necessary to obtain the best results.
- Test procedures for measuring properties of these materials should be scrutinized very carefully. The applicability of rubber standards to plastic-like materials is perhaps questionable.

#### SECTION V

### LABORATORY PROGRAM

## 1. Material Selection for Two Tire Types

The work plan for this program as presented by the AFFDL indicated that two parallel efforts (A and B) were to be undertaken. Effort A was to be directed to a one-piece cast tire, while Effort B was for a tire with cast carcass/replaceable tread. It was recognized from the beginning of the program that there would be much overlapping of the two efforts, and that it was even probable that the same material might be recommended for the carcass of both A and B efforts.

As the program has evolved, it has been found expedient to handle the two efforts simultaneously. Also, it was felt that this Phase I report should be organized and written so that the two efforts are clearly spelled out in the Conclusions and Recommendations sections only. The main text is organized as if only one effort is involved to minimize duplication. However, in the sections discussing certain specific properties, it has been pointed out where these properties affect one effort and not the other. For example, the frictional properties and abrasion resistance are of concern only to the one-piece cast tire.

Recommendations were also obtained from vendors of reinforcing fillers, particularly glass fiber. Materials used in the program were generally based on their recommendations.

## 2. Screening of Candidate Polymer Varieties

The candidate materials of construction for the two tire concepts were selected by a two-step process. Desirable polymer classes were first identified, followed by the choice of specific commercial materials within each class. Several criteria were used in making the selections, as follows:

- (1) Capable of being rotationally cast. This limited the choice to liquids or materials capable of being reduced to powders having suitable flow characteristics.
  - (2) Available in quantity from commercial sources.
  - (3) Reasonably cost competitive with standard materials now used.

(4) High strength and stiffness, since continuous reinforcement will not be used.

A review of known information quickly reduced the initial selection to four basic classes of polymer. These were

- (1) Thermoplastic polyesters
- (2) Thermoset of thermoplastic polyurethanes
- (3) Cross-linkable polybutadienes
- (4) Elastoplastics such as block copolymers of butadiene and styrene.

## 2.1 Vendor Contacts

At the initiation of the Phase I program, a major effort was made to contact vendors of candidate polymers and reinforcing fillers with four objectives in mind. These were to (1) involve the vendors in this program as much as possible, (2) obtain the best recommendations regarding specific polymers or fillers to be investigated, (3) obtain data on polymers to reduce as much as possible the property measurements which had to be made at BCL, and (4) obtain samples of the recommended polymers and fillers.

Table A-1 outlines the polymer vendors and individuals contacted. The first contact was by phone and generally enthusiastic. When a follow-up was made by letter outlining in detail what was needed for the program, the response ranged from good to very poor. It is believed that in many cases the vendors appeared uncertain about the suitability of proprietary materials. The request was frequently referred to different technical and management segments of the respective organizations.

In spite of many phone calls to follow up the original letter, several of the contacts never responded with a conclusive answer.

Data supplied by some of the polymer vendors was of limited value as discussed in the next section of this report. However, somewhat more assistance was obtained from those vendors recommending specific polymers which should be examined as part of the program. It should be noted that most of the vendors contacted did not recommend any of their polymers for the program.

## 2.2 Vendor Data

Vendors who had recommended polymers for this program were also requested to supply as much data as possible about their polymers. This was done so that the measurement of polymer properties at BCL during Phase I could be kept to a minimum. The data response by some vendors was reasonably good. On the other hand, the data which was eventually supplied by some of the vendors was somewhat disappointing for several reasons. The quantity of information supplied was very minimal, and even this was of limited value. In many cases, details were not available regarding testing conditions, which have a significant effect on some properties. Different vendors used different test conditions, which made it impossible to cross-check properties between polymers

from different vendors. For example stress-strain properties were measured at crosshead speeds ranging from 1 to 20 inches/minute.

Table A-2 is a compilation of the most pertinent data for the base polymers used in this program, and some closely related polymers. These data were taken directly from information supplied by vendors. The amount of information obtained regarding filler reinforcement, dynamic properties, and processability was negligible. Therefore, the vendor assistance was merely used as a starting point for the BCL work, and was of limited value in reducing the laboratory work which had to be done at BCL.

## 3. Selection of Candidate Polymers

The selection of specific polymers within each one of these groups was made on the basis of the vendors' recommendations, past experience at Zedron and Battelle-Columbus, and was subject to some modification as the early work progressed. Using the general guidelines suggested by Zedron for polymer stiffness, the early data indicated that the polymer, with reinforcement if used, should have a durometer hardness of 60 to 65 D. This can be readily attained with polyester and some grades of polyurethane, but is more difficult to attain with polybutadienes and the styrene-butadiene elastoplastics.

The greatest effort during this program was directed to the polyesters because of the favorable past experience by Zedron using these materials in cast tires. A 55 D durometer polyester (Polymer B) was investigated most extensively because of its hardness and melt index (18) which is favorable for rotational casting. Harder polyesters (Polymers C and D) were also examined because of the higher stiffness, although they have been reported to have a potential problem with impact resistance under some specialized conditions. A third polyester, also having a 55 D durometer hardness (Polymer E) was obtained later in the program and was briefly evaluated. This is still considered to be a development material.

Polyurethanes were also examined as a class of materials since they have a very wide range of properties depending upon the particular formulation. Two thermoset polyurethanes based on MDI and having different hardness values were examined (Polymers H and I) as well as a very hard material polyether based on TDI (Polymer J). Limited work was also done with one thermoplastic polyurethane (Polymer K) and one cross-linkable liquid polybutadiene (Polymer L).

Although elastoplastic materials were originally considered for this program, they were not actually studied in the laboratory. First, none of the vendors of these

materials recommended their use for the cast tire application. Second, a brief review of their properties indicates they do not have the strength and stiffness, and perhaps chemical resistance required for the carcass material, or cannot be processed by rotational molding.

## 4. Properties of Unmodified Polymers

The approach used to evaluate materials for their suitability to perform in a cast tire was to first characterize the unmodified polymer and relate its properties to the criteria required. If the unmodified polymer fell short of the requirements, an effort was then made to modify its properties if the shortcomings were not too severe.

## 4.1 Stress Strain Properties

The first work done was to determine stress strain properties, with the results shown in Table A-3. The properties at low elongation (yield point or below) are of particular interest since the material should not be strained beyond this point in service. Young's modulus is also very important since it is a direct measure of stiffness and a tentative minimum value of 60,000 psi has been established for this property. The data do show a good correlation between the stress at 10 percent elongation or the proportional limit

and the Young's modulus. Only one polymer (c) had a modulus approaching the desired level. It is interesting to note that the hardness, which is readily measured, approaches approximately 65 D at the desired Young's modulus.

The elongation at yield of all polymers which had a yield point was 35 percent or less, indicating that the strain in the tire will have to be kept at a low level for the polymer to be functional. Two of the polymers did not show a yield point under the test conditions. If this effect is true at other rates of strain, this may be a plus for these materials since they do not exhibit the significant permanent extension attained beyond the yield point that is characteristic of the polyesters.

Because more work was done with Polymer B than with the other polymers, two lots of material were used in the work. Since this is a relatively new material, a careful check was made on the properties of the polymer from each lot. Table A-3 shows that most properties were very similar, but that an important difference was noted in the Young's modulus. This was rerun because of the importance of this property. The Young's modulus for Lot 1 (40,000 psi) compares well with published values from the vendor, while the value measured for Lot 2 (31,000 psi) is significantly lower. This difference indicated that if this polymer is used to produce the aircraft tires, it will be necessary to

check raw materials on a lot-to-lot basis, and possibly establish a specification limit for this property.

Typical stress-strain curves for these three polymers are shown in Figure B-1.

In addition to the conventional stress-strain curves, related measurements were obtained using stress-strain cycling and stress relaxation to further characterize the polymers.

Stress-strain curves were generated in extension and retraction for three cycles to determine if hysteresis, set, and stress (Mullins) softening were significant characteristics of Polymer B. Crosshead speed was 0.02 in./min. and strain was measured directly with a strain gage. The resulting hysteresis loops are shown in Figure B-2.

It is clear that hysteresis and set are significant factors which may contribute to actual tire failures. On the first cycle, hysteresis loss amounted to about 10 percent of the area under the extension curve and decreased with subsequent cycles. Permanent set amounted to 0.5 percent for the first cycle, decreasing to 0.3 percent for the third cycle. It is not known whether, given enough time, this set would ultimately be recovered. Stress softening was not detected and this is not surprising since such softening is more typically found at high strains and in filled systems.

Descriptions of how tires made from this composition failed on test are consistent with a dynamic creep or dynamic fatigue failure mode. The heating due to hysteresis, of course, exacerbates both types of failures. For these reasons, stress-strain cycling may be a useful diagnostic tool for subsequent phases of this program.

## 4.2 Stress Relaxation

Stress relaxation and creep are measures of dimensional stability and also provide valuable theoretical information about viscoelasticity. In the former, stress is measured as a function of time with the test specimen at constant deformation. In the latter, deformation is measured as a function of time with the test specimen at constant load. The two tests are basically equivalent and supply the same information. Stress relaxation was run because it most easily accommodated existing test equipment. The test procedure is discussed in Appendix C, and the raw data are given in Table A-4 for two specimens of Polymer B and one of Compound 29-4 which is Polymer B filled with 26 phr of milled glass fiber.

Several models for stress relaxation behavior of polymers have been proposed. Two of the most useful are as follows:

$$E_t = E_1 t^{-b} \tag{1}$$

and 
$$E_t = \sum_{i=1}^{k} E_i \exp(-t/h_i)$$
, (2)

where  $E_t$  is stress at time (t),  $E_1$  is stress at unit time, b is an emperical constant,  $E_i$  and  $h_i$  are the modulus and relaxation time of Maxwell element i, and k is the number of Maxwell elements required to adequately represent the data. For the present work K=4 was found to be adequate. Of the two equations, (1) has seen most use in the literature due to the ease with which it can be fitted to the data. Of particular interest in elastomer reinforcement is the work by Sircar, Voet, and Cook (1) where the parameter, b, was related to rupture of filler/polymer bonds.

Equation (2) has been used by Bartenev and Lyalina (2) to characterize stress relaxation of reinforced elastomers. In particular, the relaxation time in the vicinity of 10<sup>4</sup> seconds was attributed to filler/polymer detachments and thus provided a means for studying the polymer/filler adhesion necessary for reinforcement. This equation has typically been fitted by a tedious graphical procedure proposed by Tobolsky <sup>(3)</sup>. Apparently, it hasn't been recognized that it can be fitted to stress relaxation data directly with the

aid of a computer and a good nonlinear least squares computer program. This was the method employed in this study and the resulting parameters are shown in Table A-5.

Instead of fitting  $E_t$  to time in Equation (2), the quantity  $E_t/E_0$ , where  $E_0$  is the initial stress, was used as the dependent variable, thus making comparison of parameters easier. Furthermore, a constant term,  $E_\infty$ , was added to the equation to allow for a finite limiting stress at very long times. With the exception of the fourth relaxation time,  $h_4$ , there was little difference between the parameters for the unfilled and glass-filled polyester.

Equation (1) can be fitted either directly with computer regression analysis or graphically with a log-log plot. Regression analysis was employed in this study with 1 added to each time measurement so that the equation would be valued at t = 0. Instead of three separate regression analyses, however, the regression was accomplished in one pass through the computer by employing the following equation:

$$\ln E_{t} = \ln E_{0} + b_{1} \ln (t + 1) + B_{1} Z_{1} + B_{2} Z_{2} +$$

$$B_{3} Z_{1} \ln (t + 1) + B_{4} Z_{2} \ln (t + 1), \qquad (3)$$

where  $Z_1$  and  $Z_2$  are dummy variables taking on values 0 and 1 only. For Polymer B (Run 1), both  $Z_1$  and  $Z_2$  were set to zero.

For Polymer B (Run 2),  $Z_1$  was set to 1 and  $Z_2$  set to zero. For Compound 29-4,  $Z_1$  was set to zero and  $Z_2$  was set to 1.  $B_1$  and  $B_2$  are the fitted regression coefficients corresponding to  $Z_1$  and  $Z_2$ . The coefficients  $B_3$  and  $B_4$  allow for difference, in relaxation rate,  $\frac{d(\ln E_1)}{d(\ln (t+1))}$ , among the three determinations.  $E_0$  corresponds to stress at zero time for Polymer B, Run 1.

Inspection of Equation (3) will reveal that this

single equation is equivalent to separate fits for the three individual data sets. The advantage of the combined regression is that significant differences in relaxation rate among the three determinations will be revealed by statistically significant coefficients  $B_3$  and/or  $B_4$ . Differences in intercept will likewise be indicated by significant coefficients  $B_1$  and/or  $B_2$ . The regression results are given in Table A-6.

The significant  $B_1$  and  $B_2$  coefficients indicate that all three intercepts (i.e. initial stresses) were significantly different, although the difference between the two Polymer B intercepts was quite small. Of most interest, coefficients  $B_3$  and  $B_4$  were not statistically significant, leading to the conclusion that no differences existed in rate of relaxation. Thus, the two equations led to similar conclusions. It appears that addition of milled glass fiber to Polymer B has not significantly changed the stress relaxation (and thus creep) behavior.

Although Equation (1) is more popular and easier to fit than Equation (2), it is not recommended because it is not valid at t = 0 unless an arbitrary constant is added to the time variable. Furthermore, by varying this constant, "unit time", the values and statistical significance of the coefficients of Equation (3) can be altered significantly. Thus, the lack of applicability of Equation (1) when t = 0 creates a problem. Equation (2), on the other hand, exhibits no such deficiency. Furthermore, since the individual relaxation times, h<sub>i</sub>, and corresponding stress elements, E<sub>i</sub>, can be shown to correspond to changes occurring in the polymer network on a molecular level, this model is more useful for interpretation of differences in stress relaxation behavior among polymer systems.

## 4.3 Miscellaneous Properties

Additional physical property measurements were made on these polymers with results shown in Table A-7. As a control, a composition representing a basic synthetic rubber tire tread compound was used. Several of the property measurements shown in Table A-7 are considered particularly significant. All polymers had a tear strength and abrasion resistance several times as great as the control, which is most desirable. Also, the heat build-up on flexing was somewhat better (lower) than the control. However, the

compression set was very high and the coefficients of friction were very low. The latter, of course, would be of concern only for the one piece tire.

One very significant difference between polymer is the glass transition temperature, which can be related to low temperature performance of polymeric materials. The polyester Polymer B had a transition temperature of -49°C, compared to 7°C and 8°C for the other two materials. This suggests that the Polymer B should have properties which are affected less by reduced temperatures in the area where an aircraft tire will have to function during winter operations.

Summarizing, the data suggest that none of the unmodified polymers meet all of the criteria. Polymer C was the only material approaching the desired Young's modulus, but had a glass transition temperature suggesting poor performance at low temperature. Other polymers performed even more poorly. Because of these results, the major emphasis of the Phase I program was shifted to the evaluation of modified polymers.

## 5. Modification of Polymers

It has been recognized from the initiation of this program that it might be necessary to modify the base polymers to obtain the desired properties. This belief was confirmed in the initial work. The two types of modification considered for this program were reinforcement and cross-linking, both

depending upon the polymer type. For example, polyesters were reinforced only, while polyurethanes were cross-linked and sometimes reinforced. Both modifications were necessary with polybutadiene.

## 5.1 Polyesters

The major effort to determine the effect of reinforcement by fillers was directed to polyesters, particularly Polymer B. Since this is more a plastic than an elastomer, consideration was first given to the type of reinforcing agents generally found effective with plastics. Materials with a high aspect ratio, such as glass fiber, asbestos, or cellulosic fiber, are commonly used. The second basic type includes materials which have a lower aspect ratio and are finely divided particulates such as silica, mica, and carbon black. Several materials in each category have been investigated.

The nature of the relationship of concentration and particle shape of the filler on modulus is illustrated by Equation (4)

$$E = E_0 (1 + 0.67fc + 1.62 f^2c^2)$$
 (4)

where E and Eo are the moduli of the filled and unfilled polymer, respectively, c is the volume fraction of filler and f is proportional to filler aspect ratio. This equation predicts that modulus will increase in proportion to the square of f and c. Among other things, this implies that small concentrations of high aspect ratio material or larger concentrations of low aspect ratio material will result in equivalent reinforcement levels. To the extent that processing considerations may impose an upper limit on either f or c, the other variable can be adjusted to achieve the desired level of reinforcement. This relationship was used to advantage in this study to achieve acceptable balances of fiber length and concentration. Of course, since filler reinforcement involves innumerable other parameters such as particle size distribution, degree of agglomeration, amount of polymer immobilized on filler surface, etc. equations such as the above are useful only in a qualitative sense and are not substitutes for actual experimentation.

### 5.2 Glass Reinforcement

Because of the highly favorable experience with glass fiber as a reinforcing agent in many other applications, it was a prime candidate for the reinforcement of the polyesters. Glass in several forms (chopped, milled, bubbles) was used, and all have been silane-treated to improve adhesion to the

polyester substrate. The chopped and milled glass had been silane-treate by the supplier so the identity of the particular silane was not known but the glass bubbles were silane-coated at BCL.

The primary work with chopped glass was done with a 3/16-inch material incorporated in loadings from 1 to 10 phr. Stress-strain data are shown in Table A-8, and graphically in Figures B-3 to B-7. To judge the extent of the reinforcement, several benchmarks were used from the early part of the stress-strain curve, such as proportional limit, stress at 10 percent elongation, stress and strain at yield, and Young's modulus.

Figures B-3, B-5, and B-6 all show the same general trend, differing only in degree. Although there is some scattering in the data, all of the three properties increased in a nonlinear manner with the glass loading. As little as 2 phr of glass essentially doubled the Young's modulus, while loadings of 10 phr tripled the value from the unfilled polymer. The effect on other properties was less pronounced.

Figures B-4 and B-7 show that the elongation at yield and break were reduced significantly by the incorporation of as little as 2 phr of chopped glass. These data suggest that the glass loading will have to be carefully balanced to maximize the advantage in improved reinforcement and minimize the loss in elongation. It should be pointed out that the data shown in Figures B-3 to B-7 were obtained on test specimens

compression molded from hot milled sheet. If this technique is used for incorporation of the filler, the sheet would have to be reduced to a powder for the material to be rotationally cast. A later section of this report shows the effect of size reduction on the properties.

Studies were also made on the use of milled glass as a reinforcing material, because Zedron has found the milled glass easier to keep dispersed during rotational casting. Three lengths of milled glass were investigated, as shown in Table A-9. The milled glass is normally used in greater quantity than chopped glass because it has an average length which is much shorter. It should be noted that a different system is used to designate length of chopped and milled glass. For example, 1/4-inch milled glass is much shorter in average length than 1/4-inch chopped glass.

Table A-9 shows the stress-strain data obtained with milled glass hot milled into Polymer B. While there is scatter in the data, there are a few trends which can be noted. Increased glass loadings and glass length increase the stress at 10 percent elongation and the yield strength, and decrease elongation at yield. There seems to be no regular pattern with respect to Young's modulus.

Because the milled glass is very hetrogeneous with respect to length, one experiment was performed in which 1/16-inch milled glass was screened into three lengths and each length evaluated. The coarsest material +200 mesh produced the best reinforcement, as seen by the stress at 10 percent elongation and yield strength. In general, the best reinforcement obtained with milled glass hot milled into the polymer was roughly equivalent to that obtained with chopped glass, although a higher glass loading was required.

In addition to hot milling the milled glass into
the Polyester B polymer, experiments were also conducted to
reinforce the polymer by powder blending. This is the
ultimate objective of the work since it produces a composite
material best suited for rotational casting. For this effort,
the Polyester B was first reduced to a 35 mesh powder by
cryogenic grinding. Several different techniques were then
tried in an effort to disperse the milled glass into the
powder. The results of this work are shown in Table A-10.
The best reinforcement, as judged by the stress at 10 percent
elongation or Young's modulus was obtained by ball milling,
a very short mixing with an Atlantic Mixer, or hard stirring
for short periods. When the powdered polymer and milled glass
were rolled together in a metal can, or a sigma blade mixer,
the result was a poor dispersion which was not tested.

These experiments clearly show that powder blending is a process which will require close control. Insufficient shear will not disperse the glass, while an excessive amount of shear will fracture the glass and decrease the reinforcement. At this time, however, the reinforcement obtained by ball milling the 1/4-inch milled glass in Polymer B powder (35-mesh) compare with the best results obtained by hot milling.

Because of the limitations of time, it was not possible to optimize the variables such as glass loading and glass length. However, the best results were obtained using the longest (1/4-inch) at loadings of 20 phr. Higher loadings are expected to reduce the elongation at yield to an unacceptable level. Limited work was done, as shown in Table A-10, to screen the glass fiber and remove the longest lengths in an effort to improve the dispersion. Results were inconclusive. The data in Table A-10 do show the good potential for using ball milling as a laboratory means of dispersing the milled glass into the polyester powder to produce a product which can be rotationally cast into a tire carcass.

Subsequent in-plant experiments conducted by Zedron on dry blending various lengths and concentrations of milled glass with a 35 mesh polyester powder by means of a high-intensity mixer indicate that this method will provide and adequately dispersed composite without evidence of particle breakage. The high-intensity mixer employs a principle of

intensive circulation wherein the material is not only driven upward, but downward as well. This forced circulation results in a uniform shear action through the charge. High velocity agitation impact combined with interparticle friction insures breakdown of agglomerates and uniform distribution of even minute quantities of reinforcement additives.

One grade of glass bubbles was also evaluated. These could not be obtained with a silane treatment for improved bonding to the polyester, so the coating was undertaken at BCL. The bubbles were coated with 0.2 phr silane A-1100 (Union Carbide) according to the vendor's instructions.

Table A-11 shows the results obtained using several levels of the glass bubbles both uncoated and silane coated. The loadings used were relatively low on a weight basis, but cover a broad range on a volume basis. The specific gravity of the glass bubbles was measured and found to be almost exactly 1/10 that of chopped glass as the bubbles are hollow. Therefore, while the loadings used are approximately the same as used for chopped glass on a weight basis, they are ten times as great on a volume basis.

Table A-11 shows no significant reinforcement with either the uncoated or coated glass bubbles. In each case, there was an increase in the Young's modulus at the highest level used. This approach to reinforcement does not look promising at this time, as additional studies would be required to develop an effective surface treatment for the bubbles.

Work was also done to evaluate three sizes of
Microglas in two polyesters. The Microglas glass was in 1/16,
1/32, and 1/64-inch sizes and was reported to be silane treated.
The two polymers supplied for this study were Polymers C and
F. Polymer C was supplied in pellet form and was reduced
to -35 mesh for powder blending, while the Polymer F that was
supplied in powder form was used as received. The Microglas
was incorporated into the polyester powder by tumbling
overnight in a metal can.

Table A-12 shows the result of this study. The most significant result is the increase in Young's modulus caused by the Microglas. With Polymer C, an increase of 60 percent was common, while a typical increase for Polymer F was in the range of 40 percent. The elongation at yield was consistently 20 percent with Polymer C compared to 30 percent with Polymer F. The lower value may not be acceptable in view of the deformations which are bound to occur in service.

The stress-strain measurements with polyester polymer reinforced by glass fiber provided few compositions with sufficient stiffness and elongation to be serious candidates for production of a cast tire. However, three of the best compositions were selected for more extensive evaluation.

This included two filled with chopped glass (2.5 and 7.5 phr) and one filled with milled glass (20 phr).

Table A-13 shows the results of the evaluations, and includes a tire tread type composition as a control. The compression set of the three compositions based on Polymer B was very uniform but high (about 65 percent) and more than twice that of the control. Rebound was 50-55 percent except for the composition containing the high level of chopped glass where it dropped to 39 percent. Tear strength and abrasion resistance were outstanding and several times that of the control composition. Heat buildup on flexing was low with the polyester compositions developing little more than half the temperature change in the control composition. This correlates with experience at the AFFDL where dynamometer tests have indicated that tires made from polyester tend to run cooler during tire tests.

The coefficient of friction of the tire material is of interest only for the one-piece cast tire. This property was measured using a silicon carbide surface and loadings of 13.5 and 20 psi on the test sample, the latter representing the upper limit of the equipment used. It is recognized that the actual pressure on the tire footprint will be much higher, estimated by Zedron at about 80 psi. However, it is believed that the relative values will be the same at both pressures. The data showed that the coefficient of friction approximately doubled when going from 13.5 to 20 psi, indicating that much higher values should be expected at 80 psi.

Table A-13 shows the values measured at 20 psi on the samples. The tire tread composition could not be measured as the friction was so high that the sample was torn from the machine. Since a high coefficient of friction is desired, particularly for a material to be used in the one-piece tire, the high loading of chopped glass produced the most desirable composition. However, even this is low compared to the tread composition. Unfortunately, the tread composition could not be evaluated under the same conditions so it is not possible to judge just how much of a difference exists between the materials. The data do suggest, however, that further investigation is needed, however, if the polyester materials are to be seriously considered for the one-piece tire.

Time did not permit the glass transition temperature to be measured on the filled compositions. However, this property is normally affected very little by filler systems, so the value for the polymer only is reported as an estimate. This should have further verification if Polyester B is still given serious consideration during Phases 2 and 3 of this program.

Since crack initiation and propagation are probable causes of tire failure, these properties were estimated using the De Mattia testing machine (ASTM D-813). This machine was designed to simulate automobile tire sidewall flexing. It subjects 1 x 6 x 0.25-inch rubber strips to bending deformations

of 180 degrees at a rate of 300 cycles per minute. The specimen has a groove running across its midportion to constrain crack growth to this area. The specimen thickness in this area is thus reduced to 0.155 inch.

Because the most promising materials being evaluated in this study are considerably stiffer than typical elastomers and yield in the vicinity of 10 percent elongation, the test machine was adjusted to achieve the lowest possible strain on the sample. With this adjustment, the travel of the moving specimen rack was reduced to 0.31 inch. The actual strain in the grooved portion of the specimens could not be calculated, but was estimated to be less than 10 percent. In the softer specimens, such as Polymer E, the strain was even less because the ends of the specimens were strained more than were those of the stiffer specimens. The resulting initiation and propagation data are given in Table A-13.

Because the test was run 24 hours a day, the number of cycles to initiation and failure could only be estimated since the test could not be monitored continuously. When crack initiation or failure did not occur during normal working hours, the initiation and failure times (cycles) were less precise and are expressed in Table A-13 as ranges.

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Data in Table A-13 indicate that Composition 34-6 containing the milled glass fiber had better resistance to flex cracking than did Composition 21-1 containing chopped glass fiber. This difference may well be due to the significant difference in length of the glass fiber. The composition containing milled fiber had a flex life approximately equal to that of the base polymer itself.

## 5.3 Nonglass Reinforcement

Work was also done to evaluate fillers other than glass fiber for reinforcing the polyester. Table A-14 shows the results obtained with two grades of fine silica, Hi-Sil 233 and Cab-O-Sil M5. The silicas were incorporated both by hot milling and powder blending. The data show that the silicas reinforced the polymer almost as well as the glass fiber when judged by the stress at 10 percent strain. However, the highest Young's modulus was 50,000 psi compared to 40,000 psi for the unfilled polymer. This is lower than many of the Young's modulus values obtained with glass fiber, and suggests that the silicas have less potential for being an effective reinforcing filler than the glass fiber.

Several other fillers are examined with the results shown in Table A-15. The best reinforcement was obtained using Calidria asbestos hot milled into the polymer. This is shown both by the stress at 10 percent elongation and Young's modulus values. The limited data indicate that the asbestos compares favorably with the glass fiber for reinforcing the

polyester resin. However, this has not been investigated further since no clear superiority is seen for the asbestos. and the increasing efforts being made throughout industry to reduce the use of asbestos wherever possible. This form of asbestos also probably could not be used in a dry blending process, as there is not enough shear to break down the fiber agglomerations.

### 5.4 Effect of Size Reduction

The initial studies to screen reinforcing materials in polyesters were accomplished by hot milling the material into the polymer and compression molding test specimens from the milled sheet. While this approach was very expedient for screening purposes, it was recognized that the milled sheet would eventually have to be reduced to a 35 mesh powder for rotational casting.

As the program progressed, steps were taken to develop a suitable procedure for the reduction of the milled sheet to a powder and to establish the effect of this size reduction on the stress-strain properties. Cryogenic grinding techniques developed for other processes were found to be very well adapted to the size reduction of polyester polymer, either nonreinforced or reinforced. The sheet material was reduced to a powder in a three-step process, as follows:

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- (1) Reduce sheet to 1/4-inch chunks in a granulator.
- (2) Reduce granulator chunks to a fine powder with a Mikropulverizer.
- (3) Screen powder from Mikropulverizer through a 35-mesh screen.

Liquid nitrogen was used to freeze the polymer to facilitate the grinding process.

The effect of the size reduction on the stress-strain properties was measured on four compositions as shown in Table A-16. For two compositions the measurements were made only on the milled sheet and the final powder. The two remaining compositions were checked at each step of the process.

The most significant effect was the loss of reinforcement, as shown by the lower Young's modulus for the molded powder.

The modulus of the reinforced material was 40,000 psi to 50,000 psi, compared to 39,000 psi for the polymer itself.

This effect was also seen in some cases by a decrease in the stress at 10 percent elongation. An opposite effect is seen in the elongation at yield. This value for the powder was generally 30 percent, essentially the same as for the polymer itself.

These data indicate this approach to processing is not practical for glass reinforcement. If the hot milled composite blend material is to be reduced to a 35-mesh powder, most of the reinforcement is lost in the size reduction, presumably due to breakage of the glass fiber.

It appears clear that the approach mentioned, in the earlier section of this report, of dry blending reinforcement particles with the polymer powder by means of a high-intensity mixer, will be required to maintain the original dimensional sizing integrity of the powder composite, in order to effect proper reinforcement.

## 5.5 Polyurethane

Thermoset polyurethane polymers are reported to have properties which make them candidates for the cast tire material. This includes high strength without reinforcement and good abrasion resistance. Based largely on the vendors' recommendations, three materials were chosen for this investigation. All required modification by cross-linking, and some effort was made to further reinforce one of the materials with milled glass.

Stress strain data for the polyurethane compositions are shown in Table A-17. Most significant is the range in the Young's modulus values of the unfilled compositions. These ranged from 14,300 to 80,000 psi. It was felt that the polymer (Material J) with the highest modulus is too hard for this application (77 D) while the other two polymers need reinforcement to increase stiffness. The unfilled polyurethanes do not exhibit a true yield point.

Table A-17 shows additional data obtained for two compositions (38-1 and 47-1) in which an effort was made to incorporate glass fiber into the polyurethane polymer before cross-linking. This was accomplished with some difficulty as the glass filled compositions were very difficult to degas, possibly due to the increase in viscosity. Although the urethane polymer was always degassed before incorporation of the glass fiber, some air was always incorporated while dispersing the glass which was very difficult to subsequently remove.

Table A-17 shows that the inclusion of glass fiber greatly increased the Young's modulus, even above the desired value. It should also be noted that the filled compositions had a definite yield point in contrast to the unfilled compositions. Other differences are of lesser importance.

Because Composition 47-1 was developed very late in the program, it was not possible to characterize it as fully as some of the other materials. Table A-17 shows that Composition 47-1 was low in rebound, and high in heat buildup and glass transition temperature compared to the other compositions. It is expected that abrasion resistance is outstanding, although time did not permit this property to be measured. Flex crack resistance and coefficient of friction cannot be estimated.

# 5.6 Polybutadiene

One of the polymer types considered for the castable tire carcass is low molecular weight polybutadiene containing reactive chemical groups which are readily cross-linked.

These materials have very low strength without reinforcing fillers, so the investigation was confined to the use of one of the most effective fillers (fine silica).

Data in Table A-18 show the results obtained with this polymer. Strength and stiffness were extremely low even with the inclusion of the fine silica. Vendors' literature showed that other modifications of the composition could be expected to produce only marginal improvement. On this basis, no further work was done with this polymer.

## 6. Preparation of Materials for Small Tire Tests

As the Phase I program developed, compositions were prepared which have stress-strain properties which are approaching the preliminary goals outlined in the beginning of the work. To verify that the goals were reasonable and to check the processing behavior of some of the compositions, a tire test of some type was desired as quickly as possible. Zedron has a small test mold which can produce a small tire to evaluate the molding characteristics of the selected compositions.

During the Phase I program, three compositions were chosen for the preparation of test tires. All were based on the use of glass fiber in Polymer B. Two contained chopped glass fiber and one was filled with milled glass fiber, as shown in Table A-19. The polymer and glass were hot milled together, and the resulting sheet was reduced by BCL, in three steps, to a 35-mesh powder which was shipped to Zedron for molding trials. Powder blends were not prepared since these could be produced with the facilities available at Zedron.

Table A-19 shows the stress-strain measurements made on the three compositions. The first two compositions (16 and 21) were evaluated only in the milled sheet and 35-mesh powder. The last composition (34) was evaluated during each stage of the size reduction. Data show a consistent pattern with respect to properties. The milled sheet had the greatest amount of reinforcement as judged by the stress at 10 percent elongation and Young's modulus. Elongation at yield was below the value of 30 percent for the polymer alone. As the material was reduced in size, the reinforcement decreased while the elongation at yield increased to approach the value for the polymer. The goal of producing a material with a Young's modulus of approximately 60,000 psi was not attained with any of the finished material, although it was reached in the milled sheet when incorporating milled glass. data suggest that higher loadings of the glass fiber might be used to attain greater reinforcement of the 35-mesh powder produced by this method.

## 7. Selection of Materials For Phase II Prototype Tires

As a part of the Phase I program it was requested that three materials be selected as potential candidates for the preparation of full-scale tires in the Phase II program. Primary consideration was given to compositions which had measured property values most closely related to the cast tire requirements as they have been defined. Consideration was also given to suitability of fabrication by rotational casting, with preference given to systems which could be powder blended or handled from a liquid rather than requiring hot milling and size reduction. Finally, the availability of materials was considered and an effort made to eliminate materials which might be objectionable such as asbestos.

Table 1 shows the final three selections. These include a copolyester elastomer, a copolyester elastomer filled with milled glass, and a thermosetting polyurethane filled with milled glass. Composition 24-5 is judged to be the most interesting of the three compositions. It can be noted that the glass transition temperature is reported as an estimate. This was the value measured for the base polymer, as the filler normally has a minimal effect on this property.

Composition 46-4 has the advantage of not requiring the mixing of a filler, but the disadvantage of having a much higher  $T_g$  value. This may have an adverse effect on low temperature performance.

Composition 47-1 was prepared very late in the program so it was not possible to check the full range of properties. It represents the best urethane composition prepared and looks favorable in all measured properties except the lower rebound value.

# SECTION VII

### PLAN FOR PHASE II ACCOMPLISHMENT

## 1. Tire Manufacturing Equipment and Methods

The basic machinery and method of manufacture to be employed to fabricate the one piece cast tire and cast carcass with a replaceable reinforced rubber tread include a rotocast machine, a tread building machine and suitable molds to form the cast tire and tread. A brief description of the equipment and process is separately provided.

### 1.1 Rotocast Machine and Process

A rotocast machine will be used to fabricate the prototype one piece cast tire and the cast carcass for the two piece replaceable tread tire. In rotational casting the hollow tire mold is located on one arm of a three arm spindle and charged with a predetermined amount of a liquid or powder The mold sections are then clamped together. The charged mold on the rotating spindle is then indexed into a preheated oven and slowly rotated about two axes simultaneously. The revolving motion forms the closed torous tire profile with desired weight and wall thickness. The desired weight of the finished closed torous tire is determined by the amount of polymer introduced into the mold cavity. When the polymer has fused into a homogeneous layer on the walls of the mold, the spindle on which the mold is mounted is then automatically indexed to the cooling station, where the mold is cooled by an automatically timed combination of forced air and water spray. Next, the spindle is indexed into the loading and unloading station, where the finished closed torous tire is removed from the mold; new polymer material introduced into the mold; and a new cycle started.

Heating Station - After the mold is charged with the liquid or powder polymer, the spindle is indexed into the oven to start the fusing process. Oven temperatures will vary over a range of 500°F to 750°F, depending on the processing characteristics of the polymer, and maintained at - 5°F, by a modulating type burner with a high velocity circulating fan and with fail safe controls. The primary requirement of the oven is to uniformly heat the hollow mold at an acceptable level and rate. The level and rate of heating is critical in the manufacture of a uniform and properly formed cast tire. Factors that need to be considered in establishing the proper heating cycle include: (1) shape of the inner mold cavity, (2) wall thickness of the closed torous tire, (3) rate of revolution of the mold, (4) ratio of rates of revolution of the two concentric rotating shafts, (5) set-point temperature of the oven, (6) physical characteristics of the powder, such as maximum particle size, particle size distribution and repose angle, (7) physical characteristics of the melt, such as viscosity, thermal diffusivity, melting temperature and latent heat of fusion, (8) time in the oven.

Cooling Station - The cooling station is a separate unit and apart from the oven station. Cooling is by an automatically timed combination of forced air and/or water spray. The method of cooling medium can be programmed for any one of five combinations. A uniform controlled cooling cycle will have a pronounced effect on the properties of the closed torous tire. The rate of cooling will affect tire shrinkage, final material density and physical properties.

Unloading/Loading Station - From the cooling station the spindle is indexed into the unloading/loading station for removal of the finished closed torous tire. When indexed, the mold is automatically locked into the correct position for unloading.

The significant feature of rotational casting is its relative simplicity, low labor cost, relative high volume output, and low investment in plant, machines and equipment. Rotational casting lends itself to automated systems for, (1) storing, transporting, and blending easy-to-handle polymer powder or liquids, and for, (2) converting the polymer into a homogeneous closed torous one piece aircraft tire or carcass. Through rotational casting, plant space requirements are reduced to a small fraction of the space required to produce an equivalent number of conventional aircraft tires or carcasses.

The rotocast machine with its separate component items is shown in Drawing D-1.

## 1.2 Reinforced Tread Building Machine and Process

An orbitread building machine will be used to fabricate the reinforced replaceable tread prior to vulcanization in a unitized sectional tread mold. The orbitread machine automatically applies extruded rubber to a sectional mandrel designed with a specific crown contour to be compatible with the carcass contour. The extruded rubber, in the form of a ribbon, is wound around the mandrel in a continuous strip. The thickness of the tread is determined by the amount of overlap from one adjacent strip to the next. The overlap is controlled by an electronic timing system which, in turn, is programmed with a program card. Each program card represents a specific tread pattern as specified for a particular tire and the mold in which it will be vulcanized. The major components of the machine are shown in Drawing E-2.

The equipment contains a water-cooled extruder and drive motor, an azimuth drive system, electrical components, pneumatic controls, and a stitcher assembly that applies the extruded ribbon to the building mandrel. The unit contains a hub for mounting the mandrel and a drive motor for rotating the mandrel during the application of rubber. Bulk rubber

in strip form is fed into the extruder screw. The extruded ribbon of rubber is then passed under the dancer arm roller, between the thickness monitoring rollers, and pressed firmly and evenly on the mandrel by rollers on the application head as the mandrel revolves. The mandrel is moved about the application head in accordance with a predetermined program which provides the proper amount of overlap from one wrap of rubber to the next. When the application of rubber is complete, the machine stops automatically. A separate cord winding attachment is provided to reinforce the tread rubber with continuous cords.

an extruded ribbon of rubber will be program wound circumferentially around the segmented mandrel and advanced progressively in an axial direction to provide a continuous overlaping gauge and width. After at least one circumferential application of the wound ribbon, a plurality of continuous reinforcing cords will be introduced at a prescribed design location in the tread between successive winds of the rubber ribbon. The overlaping relationship of the ribbon laps causes the multiple continuous reinforcing material to be disposed at a slight angle and oriented in a circumferential/helix configuration above the surface of the mandrel on which it is being wound. Before the ribbon reaches the full width of the tread, the multiple continuous cord reinforcing material

will be severed and terminated at the prescribed design location in the tread so as to be adequately covered by the rubber ribbon laps. The fabricated reinforced tread will be a circumferential/helix oriented continuous cord reinforced elastomer composite with no splices.

The orbitread method for fabricating the reinforced tread has the advantage of: (1) lower initial bulk rubber cost, (2) reduced rubber usage, (3) elimination of tread splice, (4) lower inventory requirements, (5) elimination of numerous die sizes, (6) better mold fit, (7) reduced labor cost, (8) ability to reinforce tread with continuous cord in one operation.

#### 1.3 Tread Belt Manufacture

As an alternate replaceable configuration, consideration will be given to the fabrication of a tread belt consisting of a low cord angle tread belt encompassed by a separate layer of tread rubber. The configuration will be utilized where it is deemed desirable to use wire cord reinforcement. In this construction, the manufacture of the tread belt will be separately subcontracted and the layer of tread rubber fabricated by the aforementioned orbitread method. In manufacturing the steel belt under the referenced system, there are six basic steps:

- (1) Steel wire (or stranded cable) is drawn from creel stand spools. When wire is used, several strands pass through a "curler" together, and then are threaded through a die into groups that match the strength of the stranded cable.
- (2) The curled wire (or cable) goes into the extruder/tuber.
- (3) Unvulcanized rubber is extruded around the wire (or cable) as it passes through the tuber head.
- (4) The rubber-encapsulated steel wire (or cable) is pulled onto the cooling drum assembly in strip form.
- (5) The rubber-coated steel strip is conveyed to the bias cutter where it is precisely cut to predetermined angles.
- (6) The final step is automatic butt or rubber lap splicing into a continuous belt. Precise adjustment allows uniform end count.

### 1.4 Tire Mold and Mandrel

The mold for the one piece cast tire and cast carcass will be manufactured from an aluminum alloy metal in three segments to facilitate design modification of the crown portion of the tire. For the one piece cast tire, the crown ring is designed with four circumferential ribs which will become the tread pattern of the one piece cast tire. The

cast carcass crown ring is made smooth and properly contoured to be compatible with the innerface contour of the replaceable reinforced tread.

The mandrel upon which the replaceable tread will be fabricated is designed with a contour that is compatible with the outerface crown surface of the cast carcass. The mandrel is segmented to facilitate removal after the tread building and vulcanizing is completed.

Engineering drawings for the mold and mandrel are provided in Appendix F, Drawings F-1, F-2, F-3, F-4.

### 1.5 Tread Mold

The mold for the reinforced replaceable tread will be manufactured from an aluminum alloy metal and segmented to provide a means for applying the mechanical pressure required for forming and vulcanizing the uncured reinforced rubber material. The mold will be electrically heated and contained in the load-deflection-mold press to effect vulcanization of the reinforced tread rubber material.

The tread mold is shown in Drawing G-1.

#### 2. Tire Profile

The selected initial iteration design for the 7.00 - 8 prototype cast aircraft tire has a relatively low profile with an aspect ratio of 0.65. The three primary reasons for the selected design are:

- 1. To maximize the effective use of internal air pressure to support the load and thus minimize the load required to be supported by the closed torous structure.
- 2. To distribute stresses resulting from tire deformation under load over the full crown and sidewall portion of the closed torous tire instead of having the stresses concentrated in the shoulder region of the tire.
- 3. To maximize the ground contact area of the tire footprint under given load/inflation pressure variables.

The 7.00 - 8 cast aircraft tire profile is shown in Drawing H-1.

3. One Piece Cast Tire and Cast Carcass Polymer Formulations

As stated in previous discussions, the materials selected for use in the one piece cast tire and cast carcass include a copolyester elastomer, a copolyester elastomer filled with milled glass fiber and a thermosetting polyurethane filled with a milled glass fiber. As information is gained from the testing of tires in each design iteration, consideration will be given to exploratory evaluation of various pre-polymer blends selected from the broad chemical family group of polyester and polyurethane materials.

## 4. Tread Rubber Formulation

In developing the natural rubber formulation for the tread, primary consideration was given to high speed aircraft tire performance reliability and high abrasion resistance qualities for long wear.

The selected tread rubber formulation is specified in Table I-1.

5. One Piece Cast Tire and Cast Carcass
Discontinuous Fiber and Particle Reinforcement

Previous discussion and data provided information on the improved properties of selected polymers when filled with milled glass fibers. Milled glass fibers are continuous glass filaments hammermilled into various lengths ranging from 1/32" to 1/4" which can be coated with coupling agent to establish a compatible polymer composite. Although general moldability has been established for the milled glass composite, additional work is required to be performed on the molding characteristics of composites utilizing fibers of various lengths and concentrations before an optimum length and percent concentration factor can be determined for reinforcing the one piece cast tire and cast carcass.

As a possible alternative to the use of milled glass fiber, exploratory work will be conducted on glass flakes which were not evaluated by BCL in the work undertaken in the materials survey and evaluation portion of Phase I.

Glass flakes are thin, disc like, glass elements have been hammermilled through a designated screen size and are available in particle sizes ranging from 1/64" to 1/4". Moldability of some glass flake composites has already been established in Zedron in-house programs.

From prior work accomplished on discontinuous fiber or particle filled composite material, it is quite evident that the fiber or particle needs to have a high aspect ratio for effective reinforcement. The longer and thinner the fiber — the higher the aspect ratio — the more it contributes to increasing tensile strength and Young's modulus. The high aspect ratio glass flakes should work in much the same way i.e. improving tensile and modulus.

The milled glass fibers and glass flakes will be the prime reinforcement elements in some of the tire construction iterations.

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## 6. Replaceable Tire Tread Continuous Cord Reinforcement

Extensional stiffness of the replaceable reinforced tread is a significant property as it relates to the non-derailment of the tread from the carcass when the tire is operated in a dynamic mode. High extensional modulus continuous cords represent the most acceptable tread reinforcement to maximize extensional stiffness and dimensional stability. The initial prototype replaceable tread will be reinforced with a high strength to weight ratio aramid cord. As information is gained from testing of tires in each design iteration, consideration will be given to using other cord materials such as polyester, glass and wire.

#### 7. Inflation Valve

A rubber sidewall valve has been designed for use on the one piece cast tire and cast carcass. Prior successful work accomplished by the tire industry and USAF on a sidewall valve for the 31x11.50 -16/22Pr Type VIII tire used on the F-101 aircraft strongly supports the design integrity of the sidewall valve concept.

The aforementioned sidewall valve tires were successfully qualified at a speed of 250 mph with an inflation pressure of 275 psi. Subsequent use of sidewall valves in cast tires designed and manufactured by Zedron, show a high order of reliability in the effectiveness of the sidewall valve as an inflation device.

The sidewall valve is shown in Drawing J-1.

8. Design and Construction of Initial Prototype
One Piece Cast Tire and Cast Carcass/Replaceable Tread Tire

The initial prototype one piece cast tire, in the Phase II program, will be a low profile design with an aspect ratio of 0.65 and cast from an unmodified copolyester elastomer with an approximate hardness of 65 Shore D.

The initial prototype cast carcass for the two piece replaceable tread tire, in the Phase II program, will also be a low profile design and cast from an unmodified copolyester elastomer with an approximate hardness of 55 Shore D. The replaceable tread will be formulated from a natural rubber compound reinforced with continuous aramid cords.

#### SECTION VIII

## QUALIFICATIONS

In preparing this report, the trade names of candidate polymers have not been used. This has been done because of the exploratory nature of the program and to avoid any appearance of biasing the proprietary position of individual vendors. The materials are described only by code numbers, although generic descriptions are included. Fillers and other reinforcements are specifically identified.

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**TABLES** 

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Dr. George Schael

American Cyanamid Company
Bldg 3
Bound Brook, NJ 08805

Dr. Pat Ryan
Arco Polymers, Inc.
Research Department
Glenolden, PA 19036

Dr. Glen Aliger
Central Research Labs
1200 Firestone Parkway
Akron, OH 44301

Mr. Marvin T. Kuypers

E. I. du Pont de Nemours & Co., Inc.

Nemours Bldg, Office 7516

Wilmington, DE 19810

Mr. William Gideon

<u>Eastman Chemical Products Co., Inc.</u>

P. O. Box 431

Kingsport, TN 37662

Mr. Robert Cardillo

Exxon Chemical Company
1333 West Loop S.

Houston, TX 77027

Dr. Daniel L. Kickel

B. F. Goodrich Chemical Company
6100 Oak Tree Boulevard
Cleveland, OH 44131

Mr. Robert H. Heinold <u>Hercules Polymer Technical Center</u> 800 Greenbank Road Wilmington, DE 19808

Mr. L. T. Camilleri Ruco Division Hooker Chemical Company New South Road Hicksville, NY 11802

Mr. George Pape

<u>Upjohn Company</u>

16 Pine Orchard Rd., #32

Branford, CT 06405

Mr. Walter E. Becker Director of Applications Dev. Mobay Chemical Corporation Penn-Lincoln Parkway W. Pittsburgh, PA 15205

Mr. Tom Brett
Naugatuck Chemical Company
Spencer Street
Naugatuck, CT 06770

Mr. Paul Weaver
Naugatuck Chemical Company
Spencer Street
Naugatuck, CT 06770

Mr. R. S. Hamner, Product Mgr. Phillips Chemical Company 1502 Phillips Bldg BartJesville, OK 74003

Mr. Charles Oxley
Polysar Ltd.
S. Vidal Street
P. O. Box 3001
Sarnia, Ontario, Canada N7T 7M2

Mr. Peter Glockner
Shell Development Company
P. O. Box 1380
Houston, TX 77001

Mr. Robert Sucher 90 N. Shiwassee Avenue Akron, Ohio 44313

Mr. John Miele Customer Service Thiokol Corporation Chemical Division P. O. Box 1296 Trenton, NJ 08607

Mr. L. E. Hawker

<u>Union Carbide Corporation</u>

Chemicals & Plastics

Research & Development Department

Bldg 727, P. O. Box 8361

South Charleston, WV 25303

TABLE A-2. POLYMER PROPERTY DATA SUPPLIED BY VENDORS

Property	ASTM Designation	Polymer A	Polymer B	Polymer C	Polymer D	Polymer E	Polymer G(a)	Polymer H(a)	Polymer I(a)	Polymer J(a)	Polymer K
Stress at 5% strain, psi	D-638	265(c)	1000(c)	1500(d)	2050(c)	Call N	ı-	103	•		
Stress at 10% strain, psi	D-638	420(c)	1550(c)	2000(4)	2875(c)		-	1	1	100	•
Stress at 50% strain, psi	D-638		•	•	,			eu.		7.11	1350(f)
Stress at 100% strain, psi	D-638 D-412(b)	1100(d) 925	2000(d) 2100	2500(d) 2700	(e)	1700	3030(d)	- 650	3200	2000	1500(f) 1900
Stress at 300% strain, psi	D-638 D-412	1.0	1,1	1001	1 1	2100	6120(d) -	1600	3450	11	5600(f) 4000
Tensile at break, psi	D-638 D-412	3700(d) 5950	5500(d) 500C	5700(d) 5800	6000(f)	2000	7250(d) -	7500	3500	8000	7000(f) 4800
Elongation at break, %	D-638 D-412	450(d) 800	450(d) 500	350(d) 500	395(f)	- 009	330(d)	550	350	7 292	350(f) 350
Flexural Modulus, ps1	D-790	7000	30,000	50,000	75,000	uil uil	e e e		15	80,000	120
Hardness, Shore D	D-2240	40	55	63	72	55	0.9	82A	09	75	52
Tensile Set, %	D-412	18	38	20	ı	35	20	œ	35		45
Tear Strength, pli Die C	D-624	700	900	870	1 1	1 1	1 1	1 1	1-1	1 1	900
Vicat Softening Temp, F	D-1525	234	356	363	397	338	ı	•	1	•	: 1
Melt Index, g/10 min	D-1238	2	18	7	7.5	ű.	1	1	ı	1	e F
Melting Point, F	D-2177	334	412	403	454	446	ı	ı	1	1	d
Specific Gravity	D-720	1.17	1.20	1.25	1.20	1.18	1	1	l I	1.21	4
Bashore Rebound, %	D-2632	62	53	43		8 N	45	•	ı	45	n fie
		The Party States		The second second							

Data obtained using a typical chain extender.

Smold #1 (Programia) minute. inches a 20 are at (v) All tests according to D-412 are
(c) Tested at 1 inch per minute.
(d) Test speed not given.
(e) Yields at 25% elongation.
(f) Tested at 2 inches per minute.

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TABLE A-3. STRESS-STRAIN PROPERTIES OF UNMODIFIED POLYMERS

			Stress at 10%	at 10% Proportional	Elongation at	Yield	(A)		ea.	Ham day and	S Sunox
Cemposition	Polymer	Type	psi	psi	Limit, %	psi psi	ar ireid,	psi psi		at break, hardness,	pst
<b>5-97</b>	Polymer C	Polyester	2700	2400	00	3400	25	4700	200	63	57,100
5-1	Polymer B, Lot 1	Ditto	2300	1600	7	2730	30	4000	680	99	40,000
46-3) (a)	46-3) (a) Polymer B, Lot 2 48-4)		1980	1700	∞	2550	35	4000	670	95	31,000
37-2	Polymer E	=	1000	1000	10	(b)	(9)	4400	450	56	13,300
27-8	Polymer F	2	2000	1900	6	2450	30	0097	089	55	25,000
37-7	Polymer K	Thermoplastic polyurethane	1300	1100	6	(b)	(4)	4700	510	53	14,300

Average value for two samples.

Average value for two samples. Does not show yield point when tested at 2 inches per minute. <u>a</u> <u>a</u>

3.5

TABLE A-4. STRESS RELAXATION DATA FOR POLYMER B AND COMPOUND 29-4

Polyr	mer B	Polyme			
Rur	n 1	Rui	1 2		und 29-4
Time, sec	Stress, psi	Time, sec	Stress, psi	Time, sec	Stress, psi
0	2520	0	2276	0	2538
0.15	2356	0.1	2206	0.05	2477
0.3	2247	0.25	2170	0.2	2420
0.6	2153	0.4	2147	0.35	2403
0.9	2126	0.7	2128	0.65	2379
1.2	2118	1.0	2117	0.95	2374
2.4	2093	1.6	2109	1.85	2359
4.8	2071	3.1	2088	4.85	2324
11.7	2027	4.6	2074	10.85	2291
26.7	2000	7.6	2056	19.85	2264
55.2	1973	16.6	2024	40.85	2229
175.2	1932	28.6	2000	91.85	2191
295.2	1918	49.6	1979	241.85	2141
535.2	1901	79.6	1962	391.85	2115
1015.2	1863	100.6	1947	691.85	2085
1375.2	1863	250.6	1912	1291.85	2041
1495.2	1855	400.6	1894	1891.85	2018
1855.2	1841	700.6	1874	3991.85	1970
2455.2	1836	1300.6	1849	6691.85	1936
3655.2	1808	2800.6	1815	10591.85	1905
4135.2	1808	5500.6	1794	14791.85	1883

TABLE A-5. STRESS RELAXATION DATA FITTED TO FOUR ELEMENT MAXWELL MODELS

	Polymer B	Compound 29-4
E <sub>1</sub>	0.0631	0.0575
hį	0.20	0.14
E <sub>2</sub>	0.0541	0.0549
h <sub>2</sub>	9.5	10.7
E <sub>3</sub>	0.0561	0.0609
h <sub>3</sub>	212.4	273.6
E <sub>4</sub>	0.0847	0.0854
h <sub>4</sub>		4985
$E_{\infty}$	0.74.02	0.7387
Standard Error	0.003	0.003

Note:  $E_i$  = modulus of spring in Maxwell Element, i  $h_i$  = relaxation time in Maxwell Element, i.

TABLE A-6. REGRESSION ANALYSIS FOR FITTING OF EQUATION (3) TO COMBINED STRESS RELAXATION DATA

	Coefficient	
Coefficient	Value	F Ratio
ℓnE <sub>i</sub>	7.685	
b <sub>i</sub>	-0.023	160 <sup>(a)</sup>
B <sub>i</sub>	0.028	5.8 <sup>(b)</sup>
B <sub>2</sub>	0.119	111 <sup>(a)</sup>
B <sub>3</sub>	-0.003	2.0
B <sub>4</sub>	-0.003	2.0

Overall Regression F Ratio = 187 Overall Coefficient of Determination,  $R^2 = 0.94$ Standard Error = 0.023

<sup>(</sup>a) Significant at 1% level or less.

<sup>(</sup>b) Significant at 5% level or less.

TABLE A-7. PHYSICAL PROPERTY MEASUREMENTS ON UNMODIFIED POLYMERS

Composition	Polymer	Type	Compress 10%	Compression Set 10% 25%	Rebound,	Tear Strength, pli	Pico Abrasion Resistance	Heat Buildup,	Coefficient of Friction Dry Wet	ction Wet
39-1	Tire tread (Control)	Ļ	,	27	31	220	100	32	Too high to measu	Too high to measure
5-1	Polymer B	Polyester	43	58	35	930	892	17	1.51	0.69
7-97	Polymer C	Polyester	97	89	33	1030	686	28	1.84	0.82
37-2	Polymer E	Polyester	45	79	75	750	678	•	2.35	1.07
37-7	Polymer K	Thermoplastic polyurethane	·	63	09	620	1	•	1.89	1.07
			Glass	ion		Flex Crack	Flex Cracking Resistance	ince		18
Composition	Polymer	Type	Temperature, C		Cycles to	Cycles to Crack Initiation, $c_1$		Cycles to Failure,	ailure,	0
49-1	Tire tread (Control)	ŧ,	[P\$]		12 10	1			12 20	1. 1
5-1	Polymer B	Polyester	67-		1.53 x 10 <sup>4</sup>	$1.53 \times 10^4 - 2.6 \times 10^6$ (a) 5.6 × 10 <sup>5</sup>		$>3.5 \times 10^6$ $2.7 \times 10^6$	(a)	
7-97	Polymer C	Polyester	∞		$9.8 \times 105$ (a) $\leq 9.7 \times 105$	(a)	<b>V V</b>	$<1.8 \times 106$ (a) $<1.3 \times 106$	(a)	
37-2	Polymer E	Polyester	7		>1.3 x 10 <sup>6</sup>			,		
37-7	Polymer K	Thermoplastic polyurethane	i je		<300(b)			. 25		

(a) Duplicate samples.(b) Flaw in sample.

TABLE A-8. EFFECT OF CHOPPED GLASS HOT MILLED INTO POLYMER B ON STRESS-STRAIN PROPERTIES

Composition	Glass Loading(a), phr	Direction of Testing with Respect to Grain	Stress at 10% Elongation, psi	Proportional Limit, psi	Elongation at Proportional Limit, %	Yield Strength, psi	Elongation at Yield,	Tensile at Break, psi	Elongation at Break,	Hardness, Shore D	Young's Modulus, psi
1-1	0	with	2070 2070	1700 2070	10	2600	25 30	2670 3820	360	58	40,000
3-7	0	with	2350 2330	2350	7	2600 2600	30	3650 3550	530 600	58	28,600
12-4	н	with	2970 2630	2400 2200	7	3000 2700	15 20	2870 2490	400	19	66,700
16-1	Ħ	with across	2850 2450	27.9	7	2850 2650	15 15	2900	460	62	50,000
7-5	2	with	3350 2630	3200 2500	o, o,	3350 2750	10	3350	20	62	83,300
12-1	2	with	3220 2850	3100	8 7	3220 2850	10	2550	30	63	66,700
12-6	7	with	2950 2750	2650 2300	<b>∞</b> ∞	3050 2800	10 15	2700 3000	360	62	100,000
4-1	2.5	with	3300	3200 2250	<b>σ ∞</b>	3300 2970	10 15	2550 2530	09	62	50,000
4-2	2.5	with	3150 2400	2800	10	3150 2750	20 20	2600 2520	140	62	66,700
12-3	2.5	with	3200 2870	3000	o 80	3200 2870	10	2570 2500	30	63	50,000
21-1	2.5	with across	3430 2980	3400 2700	σ. 80	3430	15 15	2720 2650	180	09	50,000
22-6	7	with	3700	3600	σ,	3800	12	2400	210	62	83,300
3-1	5	with	3350	2250 2750	66	3350 2800	10	3350 2250	10	62	100,000
3-2	٠ د	with	3570 3420	3570	10	3570	16 12	3570 2550	20	09	100,000
3-3	7.5	with	3950 3350	3920 3350	10	3920 3350	10	3920 2750	10 20	29	100,000
3-6	7.5	with	4200	4200	10 10	4200	10	4200	10	99	100,000
3-4	10	with	3900 3750	4100	90	3900	10	3900	10	. 67	100,000
3-5	10	with	3720 3770	3720 3770	10	3720 3770	10	3720 3770	10	99	100,000
m m	10	with	5010 3300	5010 2500	10 7	5010 3310	10	5010	10	62	80,300

(a) Glass used was OCF 419AA-3/16.

TABLE A-9. STRESS-STRAIN PROPERTIES OF COMPOSITIONS CONTAINING MILLED GLASS FIBER HOT MILLED INTO POLYMER B

	Filler		Direction of	Stress at 10%	Proportional	Elongation at	Yfeld	Flonsation	Tongila	Floroarton		Variet
Composition	п. Туре	Loading,	Testing with Respect to Grain	Elongation, psi	Limit,	Proportional Limit, %	Strength, psi	at Yield,	at Break,	at Ereak,	Harchess, Shore D	Modulus,
1-1	None	0	with	2070	1700	8 5	2600	25	2670	360	Ŋ	40,050
		;				2	0/67	2	0707	000	•	78,500
13-6	OCF P1178-1/4	10	with	2750	2200	7	2950	20	2830	310	61	100,000
			across	7650	1750	<b>8</b> 0	2750	15	2600	270	,	66,730
22-1	OCF P117B-1/4	15	with	3000	2500	00	3200	15	2500	100	9	50,600
13-7	OCF P117B-1/4	20	with	3400	3250	6	3400	10	2600	30	19	65.700
			across	2820	2400	<b>60</b>	2800	10	2450	50		50,000
33-2	OCF P1178-1/8	20	with	2700	2700	10	2700	10	2500	30	65	50,000
1-2	OCF P117B-1/16	10	with	2360	2020	80	2650	20	2450	140	09	\$0.00
			across	2500	2380	80	2650	20	2430	130	•	50,000
7-6	OCF P117B-1/16	25	with	3150	2820	6	3150	10	3150	10	67	65,700
			across	2770	2000	80	2770	10	2770	10	•	65,700
10-1	OCF P1178-1/16 (-100 mesh	25	vith	3570	3570	10	3570	10	3570	10	63	66,700
	+zoo mesu		across	2800	2800	10	2800	10	2800	10		66,700
10-2	OCF P1175-1/16 (-200 mesh)	25	with	2950	1820	9	2950	10	2670	20	09	100,000
	(+375 mesh)		across	2500	1750	7	2500	10	2500	10		66,790
10-3	OCF P117B-1/16 (-325 mesh)	25	with	2670	2300	80	2670	10	2370	07	62	50.003
			across	2650	2050	80	2650	10	2350	07		66,730

TABLE A-10. STRESS-STRAIN PROPERTIES OF COMPOSITIONS CONTAINING MILLED GLASS POWDER BLENDED INTO POLYMER B

	Glass Fiber			Stress at 10%	Proportional	Elongation at	Yleid	Elongation	Tensile	Elongation		s Strex
Composition	Туре	Loading, phr	Blending	Elongation, psi	Limit, psi	Proportional Limit, %	Strength, psi	at Yield,	at Break,	at Break,	Hardness, Shore D	
5-1	None		ı	2300	1000	7	2730	30	7000	680	36	40,500
24-1	OCF 2117B-1/16	25	Rolled 24 hrs in metal can			Poor	Poor dispersion,	i, no test -				
24-2	OCF P1178-1/16 (+200 mesh)	25	Ditto			ļ	- Pitto					
24-3	OCF P117B-1/4	20	=	Ì			<u> </u>					
30-1	OCF P117B-1/4 (-20 mash)	50	Rolled 1 hr fn netal can				=					
31-1	OCF P117B-1/4	20	30 minutes in ball mill				<u>.</u>					
24-4	Dicto	20	l hour in ball mill	2600	2200	7	2600	15	2300	20	62	66,700
24-5	The state one state as	20	2 hours in ball	2600	1700	7	2680	15	2430	80	62	65,703
24-6	State City The Bull	20	4 hours in ball mill	2500	1900	∞	2650	20	2450	210	61	66,700
31-2		20	2 minutes in Atlantic Mixer	2400	1600	7	2550	15	2400	07	09	56,700
5-97		20	15 minutes in Atlantic Mixer	2500	2100	6	2650	20	2500	200	62	57,100
26-2	MA-MITTERS	20	30 minutes in Atlantic Mixer	2300	2200	10	2420	20	2430	240	62	57,100
30-2	r	20	30 minutes in Signa Blade Mixer	87		Poor	Poor dispersion, no test	no test				
31-3	OCF P1178-1/15 (-20 mash)	20	Hand-stirred for 2 minutes	2400	1800	7	2500	15	2300	70	62	66,700

TABLE A-11. STRESS-STRAIN PROPERTIES OF COMPOSITIONS CONTAINING GLASS BUBBLES POWDER BLENDED INTO POLYMER B

	Filler		Stress at 10%	Propertional	Elongation at	Yfeld	Elongation	Tensile	Flongation		Vound
Composition	Type	Loading, phr	Elongation, psi	Limit, psi	- 1	Strength, psi	at Yield,		at Break,	Hardness, Shore D	Modulus, ps1
33-1	None		1900	1900	10	2600	30	4500	730	56	30,800
1-97	B-30-B (uncoated)	2.2	2250	2200	ω	2600	30	2700	230	57	33,300
7-84	Ditto	S	1800	1700	6	2300	30	2250	06	57	33,300
48-3	The second second	7.5	1800	1800	10	2250	30	2200	20	09	33,300
52-1	ACCUPATION OF THE PERSON OF TH	10	1900	1350	7	2150	30	2100	70	58	007,44
52-4	B-30-B (silane coated)	1	2200	1850	80	2550	30	2900	410	09	30,800
52-5	Ditto	2	2000	2000	10	2550	30	2700	360	09	30,800
52-6	School S	2	2100	1800	co	2450	20	2300	70	- 19	40,000

TABLE A-12. EFFECT OF USING GLASS FILLER SUPPLIED BY ZEDRON AS A REINFORCING FILLER FOR POLYESTER

		Glass Filler	Loading	Stress at 10% Florostion	Proportional	Flongation at	Yield	Elongation	Tensile	Elongation		Young's
Composition	Polymer	Type	phr	psi	psí.	Limit, %	ps1	at rieid,	at break,	at Break,	Hardness, Shore D	Modulus, psi
27-1	Polymer C	1	0	3300	2300	7	3800	25	4100	510	79	66,700
27-2	Ditto	Microglas-1/16	S	3100	2000	7	3450	25	4300	420	99	80,000
27-3	Ŀ	Microglas-1/16	15	3200	3100	σ	3000	20	3200	07	62	80,000
27-4		Mcroglas-1/52	5	3200	2000	7	3700	20	3000	110	99	99,700
27-5	2	Microglas-1/32	15	3100	2500	00	3300	20	3900	80	65	80,000
27-6	:	Microglas-1/64	5	2900	2000	7	3300	20	2900	80	65	80,000
27-7	2	Microglas-1/64	15	2900	2100	80	3250	20	2800	70	29	80,000
27-8	Polymer F	1	0	2000	1900	σ.	2450	30	0095	680	55	25,000
27-9	Ditto	Mcroglas-1/16	s	2050	2050	10	2400	30	3900	520	57	30,800
27-10		Mcroglas-1/16	15	2200	1700	89	2350	30	2850	370	56	40,000
27-11	:	Microglas-1/32	5	2100	1300	7	2400	30	4000	520	57	40,000
27-12		Microglas-1/32	15	2100	1900	œ	2350	20	2900	370	59	50,000
27-13		Microglas-1/64	5	2000	1450	œ	2300	30	3800	530	57	36,400
27-14		Microglas-1/64	15	1900	1400	æ	2200	07	3000	420	57	44,400

Note: Both polymers and fillers supplied by Zedron. Mixing was by powder blending in metal can on slow rolls.

TABLE A-13. PHYSICAL PROPERTY MEASUREMENTS ON MODIFIED POLYMERS

Composition	Polymer-Filler System	Compression Set 10% 25%	ion Set 25%	Rebound,	Iear Strength, pli	Pico Abrasion Resistance	Heat Buildup,
39-1	Tire tread (Control)	ŀ	27	31	220	100	32
21-1	Polymer B + 2.5 phr 419AA-3/16 chopped glass fiber	•	65	55	940	924	16
32-4	Polymer B + 7.5 phr 419AA-3/16 chopped glass fiber	89	63	39	670	986	17
34-6	Polymer B + 2C phr P117B-1/4 milled glass fiber	ı	79	20	250	724	20
36-1	Polymer I (unfilled but crosslinked)	ı	7,	27	650	317	53
47-1	Polymer I with 20 phr Pll7B-1/4 milled glass fiber and crossithied		55	13	630	· F	35
4 - 5 M		Coefficient		Glass	Flex Cracking Cycles to Crack	Rest	stance Cycles to
Composition	Polymer-Filler System	Dry Wet		lemperature, C	initiation,		Failure, Cr

				Glass	Flex Cracking Resistance	sistance	42
mpost tion	Polymer-Filler System	Coefficient of Friction Dry Wet	cient ction Wet	Transition Temperature, C		Cycles to Failure, C <sub>t</sub>	0.2
39-1	Tire tread (Control)	Too	Too high to measure		Ba-	za E	
21-1	Polymer B + 2.5 phr 419AA-3/16 chopped glass fiber	1.71 1.07	1.07	-49 (est)	1.5 x 10 <sup>4</sup>	3.0 × 10 <sup>4</sup>	
32-4	Polymer B + 7.5 phr 419AA-3/16 chopped glass fiber	2.51	1.28	-49 (est)			
34-6	Polymer B + 20 phr P117B-1/4 milled glass fiber	1.17	1.07	-49 (est)	≤9.7 × 10 <sup>5</sup>	≤1,3 × 10 <sup>6</sup>	
36-1	Polymer I (unfilled but crosslinked)	1.28	0.97		Section 14 Acres		
47-1	Polymer I with 20 phr Pil7B-1/4 milled glass fiber and crosslinked	1	11	16	,	· •	

TABLE A-14. EFFECT OF USING FINE SILICAS AS A REINFORCING MATERIAL FOR POLYMER B

Composition	n Filler	Loading,	Stress at 10% Elongation, psi	at 10% tion,	Proportional Limit, psi	Elongation at Proportional Limit, %	Yield Strength, psi	Elongation at Yield,	on Tensile I, at Break, psi	e Elongation k, at Break,	Hardness, Shore D	Young's Modulus, psi
						Hot Milled	lied					
1-1	None	ij	2070		1700	∞	2600	35	2670	360	58	40,000
22-4	H1-Sil 233	S	2300		2300	10	2850	25	2500	70	19	40,000
22-5	Dirto	10	2700		2400	æ	3050	20	2700	30	62	007,44
7-3	=	10	2900		2300	80	3070	20	3050	20	19	50,000
13-8	Cab-0-S11 M5	2	2700		2350	80	2850	25	3200	067	09	50,000
13-9	Ditto	S	2500		1950	7	2850	30	3370	580	19	50,000
22-2	=	7.5	2500		2100	σ	3020	30	2700	130	19	50,000
27-3	F	10	2600		2000	တ	3150	30	2700	220	62	20,000
						É	7					
						rowder blend	prend					
5-1	None	-	2300		1600	7	2730	30	4000	680	95	40,000
19-2	Hi-Sil 233(a)	10	2700		2500	œ	2700	15	2500	20	09	50,000
37-1	Cab-0-Sil M5(b)	5	2200		2200	10	2750	30	2600	100	57	33,300
37-4	Ditto	10	2300		1900	80	2650	20	2300	30	65	40,000
2-52	Cab-0-S11 M5(a)	0 10	2600		2500	6	2900	20	2600	30	63	50,000
19-3	Ditto	5	2600		2000	7	2850	26	2200	14.0	0.9	000

(a) Polymer and silica blended together 24 hours in metal container on slow mixing rolls.

(b) Polymer and silica blended 4 hours in ball mill.

TABLE A-15. STRESS-STRAIN PROPERTIES OF POLYMER B REINFORCED WITH MISCELLANEOUS FILLERS

	Filler		Stress at 10%	at 10% Proportional	Elongation at	Yield	Elongation	Tensile	Elongation		s, Sunox
Compesition	Type	Loading, phr	Elongation, psi	Limit, psi	Proportional Limit, %	Strength, psi	at Yield,	at Break, psi	at Break,	Hardness, Shore D	Modulus, 251
					Hot Milled	Pal					
7-1	Calidría Asbestos HPP	5	2830	2150	œ	2950	20	2650	20	09	66,700
1-5	Calidria Asbestos HPP	10	3250	2650	80	3300	15	2800	30	62	100,000
4-3	Santoweb H	10	2450	2200	80	2570	20	2400	20	57	33,300
4-4	Santoweb W	10	2200	1450	7	2350	20	2300	100	28	50,060
3-9	Aluminum flake	v	2250	1250	9	2500	20	2420	30	27	66,700
3-8	Aluminum flake	10	1900	1700	80	2070	10	1800	20	57	50,000
7-2	N-330 Carbon Black	10	2030	1900	80	2030	10	2030	10	09	20,000
10-4	Micronized talc	10	2500	1950	80	2650	20	2500	160	58	50,000
10-5	Calcene IM	10	2370	1750	œ	2370	10	2370	10	09	50,000
					Powder Blend	pu			2		
37-5	Calidwia Asbestos HPP	10	2050	1450	7	2050	10	2050	10	62	50,000

TABLE A-16. EFFECT OF SIZE REDUCTION ON STRESS-STRAIN PROPERTIES OF POLYMER B COMPOSITIONS

16-1   1 phr Oct 419Aa-3/16   Hilled sheet   2400   1500   6   2650   20   3000   3000   21-1   2.5 phr Oct 719Aa-3/16   Hilled sheet   3430   3400   9   2430   15   2720   2200   22-1   2.5 phr Oct 719Aa-3/16   Hilled sheet   3430   3400   3500   8   2730   3700	Composition	Loading	Description of Material	Stress at 10% Elongation, psi	Proportional Limit psi	Elongation at Proportional Limit, %	Yield Strength, psi	Elongation at Yield,	Tensile at Break, psi	Elongation at Break,	Hardness, Shore D	Young's Modulus, psi
Ditto -35 mesh powder 2400 1550 6 2650 30  2.5 phr OCF 419Aa-3/16 Milled sheet chopped glass  Ditto -35 mesh powder 2400 2200 8 2730 30  7.5 phr OCF 419Aa-3/16 Milled sheet through glass  Ditto granulator  "	1-91	1 phr OCF 419AA-3/16 chopped glass	Milled sheet	2350	1400	9	2650	20	3000	410	09	55,600
2.5 phr OCF 419AA-3/16 bitto       Milled sheet       3430       3400       9       2430       15         Ditto	1-91	Ditto	-35 mesh powder	2400	1550	9	2650	30	2950	087	62	50,000
Ditto         -35 mesh powder         2400         2200         8         2730         30           7.5 phr OCF 419Ad-3/16 chopped glass         Milled sheet through         3900         3500         8         3920         15           Ditto         Xilled sheet through         3500         2800         8         3500         15           "         Through Mikropulverizer         2500         2400         10         2850         20           20 phr OCF P117b-1/4         Milled sheet         2650         1550         6         2550         15           20 phr OCF P117b-1/4         Milled sheet         2650         1750         6         2650         15           20 phr OCF P117b-1/4         Milled sheet         2650         1750         6         2650         15           milled glass         Through Mikropulverizer         2400         2050         8         2600         20           "         "         "         "         2650         2550         25         25	21-1	2.5 phr OCF 419AA-3/16 chopped glass	Milled sheet	3430	3400	6	2430	15	2720	180	62	000 09
7.5 phr ocF 419AA-3/16 milled sheet chrough glass         Milled sheet through granulator         3500         2800         8         3920         15           Ditto         Milled sheet through granulator         2500         2300         8         3500         15           "         "Itrough Mikropulverizer         2400         2400         10         2850         30           20 phr oCF P173-1/4         Milled sheet         2650         1550         6         2700         15           20 phr oCF P173-1/4         Milled sheet         2600         1750         6         2650         15           milled glass         Milled sheet through Mikropulverizer         2400         2650         2650         15           ""         Through Mikropulverizer         2600         1750         6         2650         2650           ""         Through Mikropulverizer         2400         2650         2650         2650         2650           ""         Through Mikropulverizer         2400         2650         8         2600         26           ""         ""         ""         ""         ""         ""         ""         ""           ""         ""         ""         ""         ""	21-1	Ditto	-35 mesh powder	2400	2200	00	2730	30	3100	200	09	50,000
Ditto         Milled sheet through granulator         3500         2800         8         3500         15           "         Through Mikropulverizer with 0.187-inch screen         2500         2400         2400         10         2850         30           20 phr OCF P117B-1/4 Milled sheet         Milled sheet         2650         1550         6         2700         15           Phitto         Milled sheet through glass         2600         1750         6         2650         15           "         Through Mikropulverizer with 0.187-inch screen         2400         2050         8         2600         20           "         Through Mikropulverizer with 0.187-inch screen         2250         25         25	32-1	7.5 phr OCF 419AA-3/16 chopped glass		3900	3500	60	3920	15	3920	20	29	100,000
## Through Miled sheet 2500 2300 8 3070 20  **Through Miled sheet 2650 1550 6 2700 15  **Through Mikropulverface 2650 1750 6 2650 15  **Through Mikropulverface 2600 1750 6 2650 15  **Through Mikropulverface 2600 1750 6 2650 15  **Through Mikropulverface 2600 1750 6 2650 20  **Through Mikropulverface 2600 1750 6 2650 20  **Through Mikropulverface 2600 2050 8 2650 20  **Through Mikropulverface 2600 2050 8 2650 20	32-2	Mtto	Milled sheet through granulator	3500	2800	00	3500	15	3500	20	19	99,700
20 phr OCF P1178-1/4 Milled sheet 2650 1550 6 2700 15 milled glass milled glass  Ditto Milled sheet through 2600 1750 6 2650 15 granulator  " Through Mikropulverface 2400 2050 8 2600 20 with 0.187-inch screen 2250 1900 6 2550 25	32-3		Throngh Mikropulverizer vith 0.187-inch screen	2500	2300	60	3070	20	2600	20	09	74,400
20 phr OCF P1178-1/4 Milled sheet 2650 1550 6 2700 15 milled glass Ditto Milled sheet through 2600 1750 6 2650 15 grapulator Through Nikropulverface 2400 2050 8 2600 20 with 0.187-inch screen 2250 1900 6 2550 25	32-4	The state of the state of	-35 mesh powder	2400	2400	10	2850	30	2550	140	09	20,000
### Milled sheet through 2600 1750 6 2650 15 graphulator	34-1	20 phr OCF P1175-1/4 milled glass	Milled sheet	2650	1550	9			2370	20	58	65,700
" Through Mikropulverizer 2400 2050 8 2600 20 vith 0.187-inch screen 2250 1900 8 2550 25	7.77	Ditto	Milled sheet through granulator	2600	1750	9	2650	ກ	2350	8	9	57,000
-35 mesh powder 2250 1900 8 2550 25	34-3	The services and	Through Mikropulverizer with 0.187-inch screen	2400	2050	60	2600	20	2400	160	9	20,000
	34-6		-35 mesh powder	2250	1900	40	2550	22	2400	280	19	\$9,000

TABLE A-17. STRESS-STRAIN PROPERTIES ON URETHANE COMPOSITIONS

Young's Modulus, psi		28,600	133,000	99, 700	14,300	15,400	11,890	1,100	80,000		14,300							
Hardness, Shore D		65	99	67	50	0.7	36	32	11	Z I A	53	NIATO RESERV						
Elongation at Break,		330	20	50	510	430	20	520	07		510	ligd			37-7	1	•	
Tensile E at Break, psi		2400	2900	3200	0009	3000	750	0555	4300		4700	rau .			1 43-1	al.		100
Elcngation at Yield, at	rethane	(a)	15	70	(a) (a)	(a)	(a)	(a) (	(a) 7	Urethane	(a) /			by weight	25-1 9-1		0	•
Yield E Strength, psi	Thermoset Urethane	(a)	3000	3250	(a)	(a)	(a)	(a)	(a)	Thermoplastic Urethane	(a)	151 - 9116.2		٦	-1 25-2		00 100	
Elongation at Proportional Limit, %		ω	7	œ	∞	80	80	80	∞	H	6		Recipe	Ingr	47-1 20-	100 -		1
Proportional E Limit, psi		1600	2200	2300	200	200	200	150	3200		1100	es per minute.			36-1 38-1	100 100		1
Stress at 10% Elongation, psi		1800	2800	2700	006	700	700	170	3800		1300	(a) No yield point when testing at 2 inches		į	1610n			
Base Polymer		Polymer I	Ditto	=	Polymer H	Ditto	*	•	Polymer J		Polymer K	point when te		C	Composition	Polymer I	Polymer II	rolymer J
Composition		36-1	38-1	47-1	20-1	25-2	25-1	9-1	43-1		37-7	(a) No yield						

a	)
j	
C	,
Ď.	

			In	gredient	Ingredients, parts by weight	by weig	ht	90	
Composition	36-1	38-1	47-1	20-1	25-2	25-1	9-1	43-1	37-7
Polymer I	100	100	100	,	1		ř,		ľ,
Polymer II	,	1	•	100	100	100	100	1	,
Polymer J	ı	ı	1	ı	4		•	100	í
Polymer K	•	,	ı	1	1		1		100
Multrathane XA	19	19	19	15.2	19.0	22.8	1		p p
Butanediol	ı	,	ı	1	ı	,	6.5	d	1
Trimethylol propane	1	ı	ı	,	,	,	9.0	•	ä,
Plurocol TP-440	2.1	2.1	2.1	ı	,	1	1	1	1
PPG 3531-1/8 chopped glass	•	10	ı	•	ı	•	ı	1	ì
OCF P117B-1/4 milled glass	ı	1	20	1	ī	•	•	ī	ı
MOCA	•		•	ı		•	ı	25.5	ł

TABLE A-18. STRESS-STRAIN PROPERTIES OF A POLYBUTADIENE COMPOSITION

Properties	Composition 15-1
Stress at 10% Strain, psi	50
Tensile at Break, psi	220
Elongation at Break, percent	150
Hardness, Shore D	9
Ingredients	Parts by Weight
Polymer L	100
Hi-Sil 233	50
Dibutyltin dilaurate	0.05
Toluene-2,4-diisocyanate	7.1
Mile PA III.	

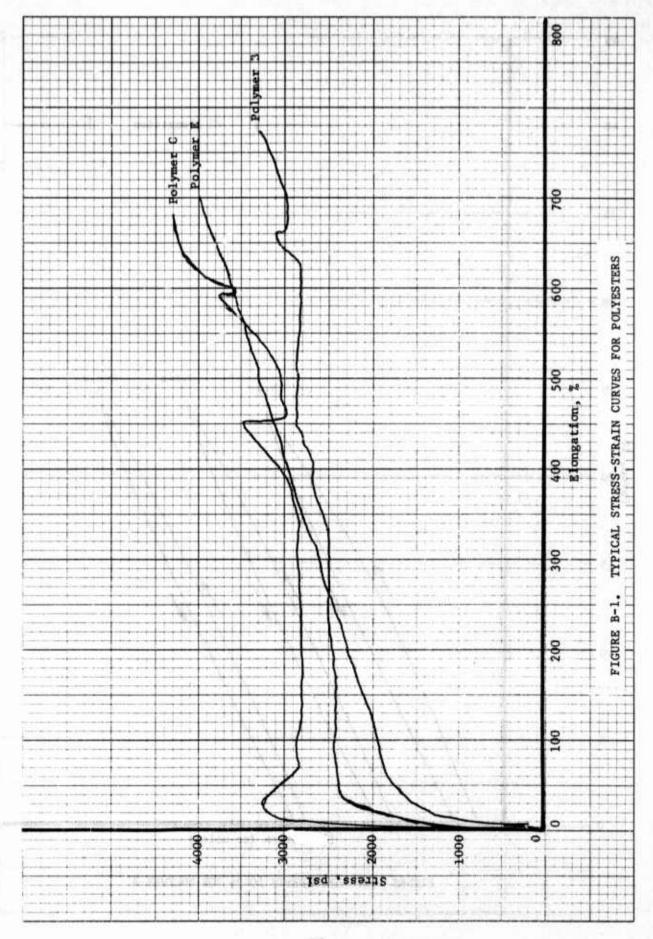
TABLE A-19. STRESS-STRAIN PROPERTIES OF MATERIALS SHIPPED TO ZEDRON FOR PREPARATION OF TEST TIRES

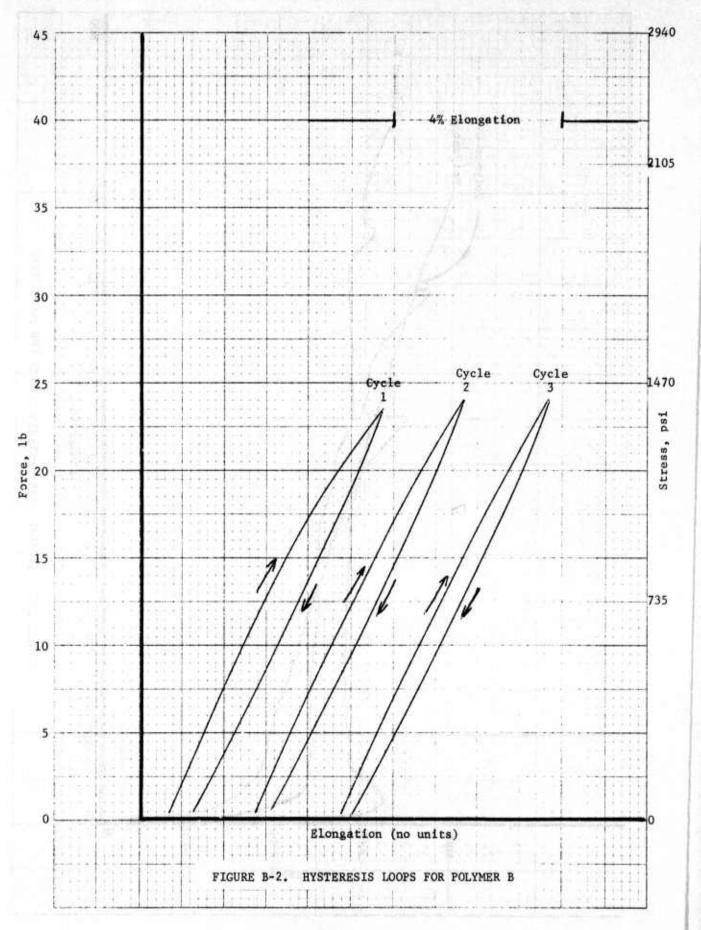
Composition	Filler Loading	Stress at 10% Elongation, psf	Proportional Limit, psi	10% Proportional Elongation at Yield on, Limit, Proportional Strength, psi Limit, 7 nei	Yield Strength,	Elongation Tensile Elongation at Yield, at Break, at Break,	Tensile at Break,	Elongation Tensile Elongation Young's at Yield, at Break, at Break, Hardness, Modulus,	Hardness,	Young's Modulus,
1-91	1 phr OCF 419AA-3/16 chopped glass	2400	1550	9	2650	30	2950	7 780	Shore D	50,000
21-1	2.5 phr OCF 419AA-3/16 chopped glass	2400	2200	8 XIX	2730	30	3100	200	09	50,000
34-6	20 phr OCF P1178-1/4 milled glass	2250	1900	IWRQ ©	2550	25	2400	280	19	50,000

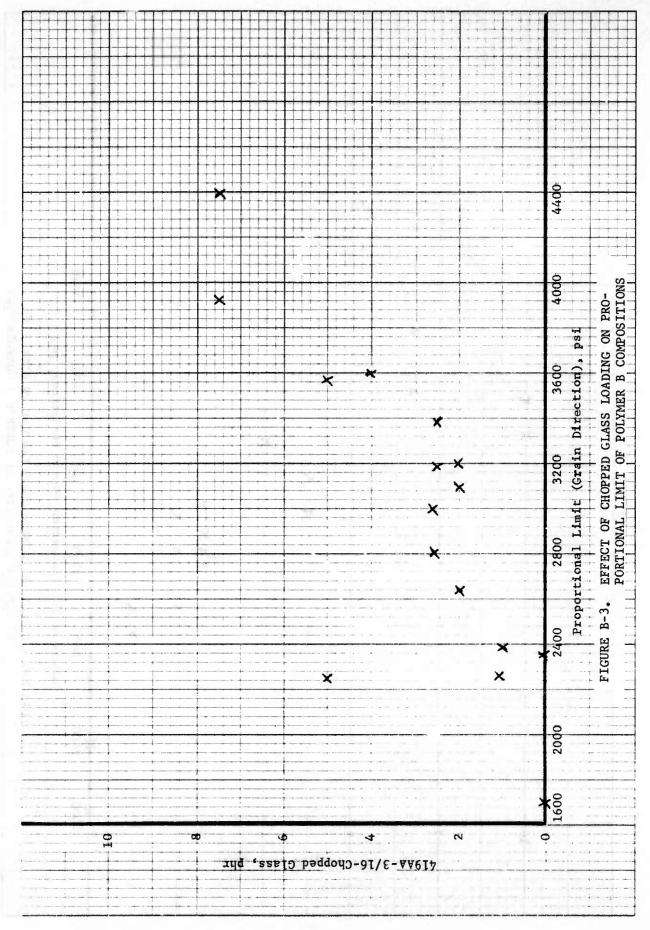
Note: All materials shipped as -35 mesh powder.

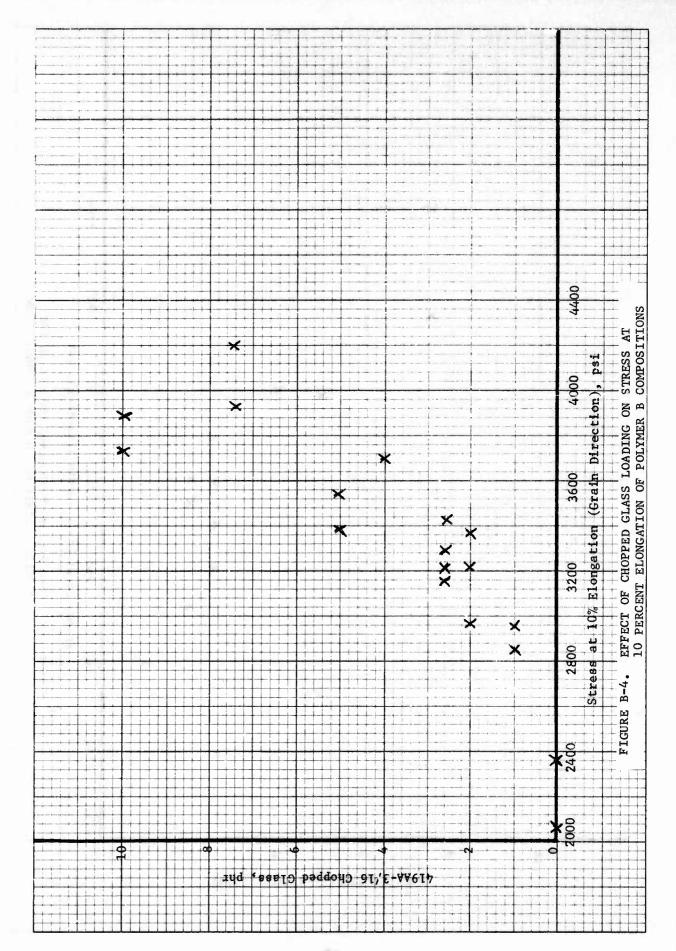
APPENDIX B

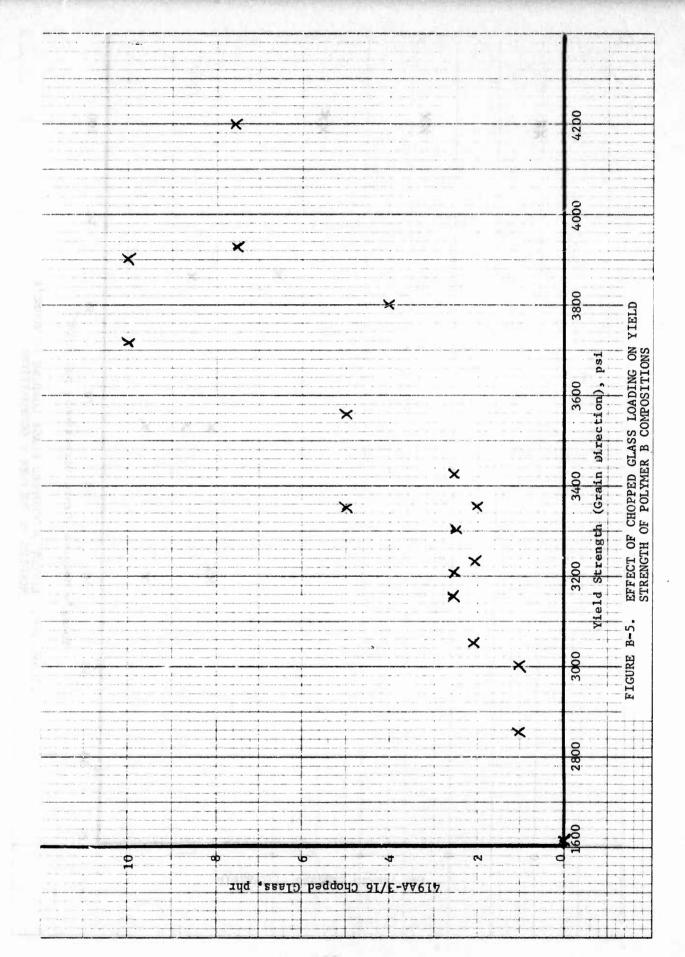
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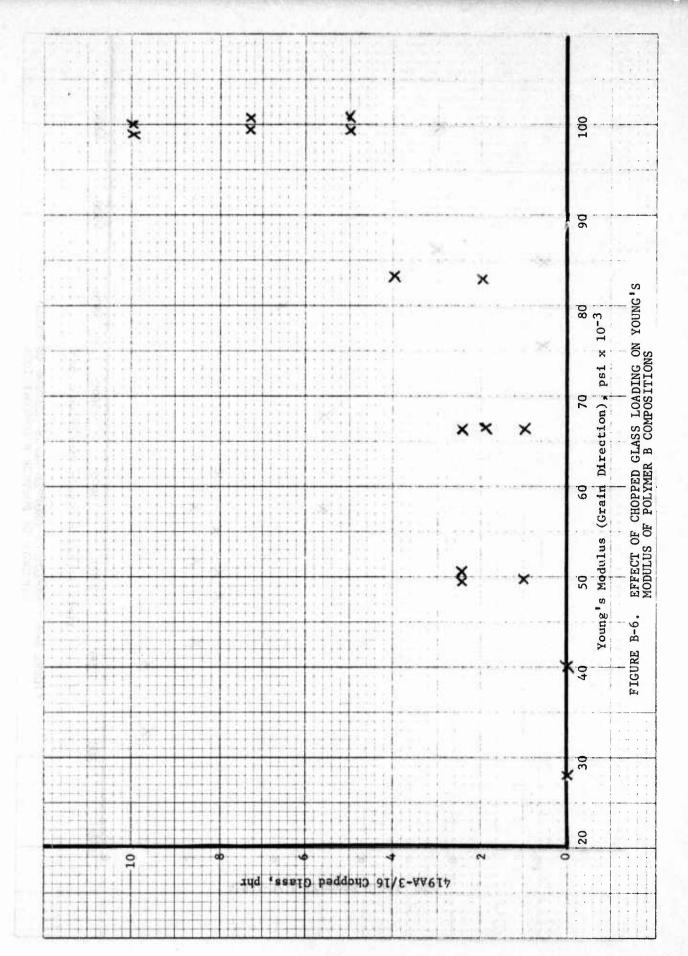


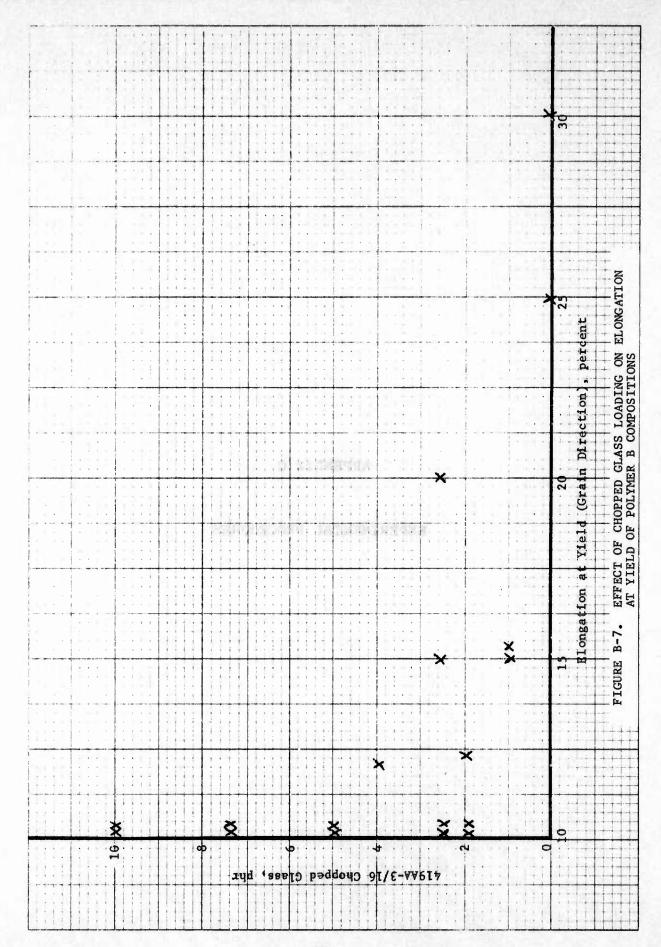












APPENDIX C

EXPERIMENTAL PROCEDURES

#### APPENDIX C

# EXPERIMENTAL PROCEDURES

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This section describes the experimental procedures used throughout the Phase 1 program. This includes both sample preparation and the measurement of property values. Because the thermoplastic polyesters are relatively new materials and are quite different in many ways from existing materials, it was frequently necessary to modify standard procedures and adapt them to the specific requirements of the polyesters. Detailed procedures follow.

## Sample Preparation

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Polyesters

The polyesters as received were all in pellet form. To incorporate reinforcing fillers, the pellets either had to be hot milled or reduced to a powder for powder blending. Hot milling was done on an electrically heated two-roll mill, with the rolls heated to 410°F. The pellets were placed in the nip of the stationary mill and allowed to warm up for 1 minute. The mill was started and the hot milling continued for 3 minutes. Fillers were added as soon as a continuous band of polymer had formed on the mill (about 30 seconds). At the end of 3 minutes, cooling water was turned on and the batch cooled. After an additional 2 minutes, the batch was firm enough to cut from the mill roll.

When powder blending was to be employed, the pellets were chilled in liquid nitrogen and cryogenically ground in a Mikropulverizer through a 0.010-inch screen. The powdered polymer and fillers were mixed together in one of several ways, as described in an earlier section of this report.

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When polymer and filler were blended, test plaques were compression molded using a 5-inch-diameter positive displacement mold in an electrically heated Preco press.

Materials were heated for 10 minutes at 410° F under 30,000 pounds of pressure. After the heating, the materials were cooled to about 320° F and removed from the mold. Flat plaques about 0.070 inch thick were used for all stress-strain, hardness, tear strength, and friction tests. Special shapes had to be molded for compression set, heat buildup, and flex cracking.

## Property Measurements

Stress-Strain (ASTM D-638)

Stress-strain curves were determined using dumbbell test specimens (Die C, ASTM D-412) pulled at 2 inches per minute. Elongation was determined using 1-inch bench marks and a manual pipper to mark the stress-strain curve.

Since several properties were determined from the stress-strain curve, these are defined as follows:

Line City and more from an reading artis

Stress at 10 Percent Strain. The 10 percent elongation point was marked on the curve with the pipper, and the stress noted at that point.

distance struck the westing bounces in nexture

Proportional Limit. Stress and strain at the point where the stress-strain curve deviates from the initial straight line.

Yield Point. Stress and strain where stress-strain curve first has a zero slope.

Young's Modulus. Initial slope of stress-strain curve.

Compression Set (ASTM D-395)

This measurement was made according to ASTM D-395 except that two compression values were used. The test is most commonly run by compressing the test specimens 25 percent. Since this introduces strains well beyond the yield elongation in most of the materials, a lower compression value of 10 percent was also used. Compression was for 22 hours at 158 F, after which the samples were removed and the set measured.

Resilience (Rebound) (ASTM D-2632)

Resilience was measured using a Bayshore Rebound

Tester. In this instrument, a steel weight is dropped on
a sample of the test material and the percentage of the drop
distance which the weight bounces is measured.

Tear Strength (ASTM D-624)

Tear strength was measured on test specimens cut using Die C. The test specimens were pulled at 2 inches per minute using a Scott L-6 Model tester.

Abrasion Resistance (ASTM D-2228)

The relative abrasion resistance was measured using a Pico Abrader. Flat molded sheets from the materials under evaluation were bonded to test specimens from earlier Pico tests to obtain adequate clamping in the test jig. Abrasion tests were made according to the ASTM procedure.

Flex Cracking (ASTM D-813)

Resistance to flex cracking was determined using the DeMattia machine and test specimens which had not been pierced.

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Heat Buildup (ASTM D-623)

This measurement was made using a modified Goodrich Flexometer. As the machine is traditionally run, the sample deflection under a static load of 143 psig and a cyclic stroke of 0.175 inch is adequate to maintain the samples securely in the machine. Because the materials currently being examined are so much stiffer than materials traditionally examined, the static loading on the test specimen was increased at 250 psig and the cyclic stroke decreased to 0.050 inch. Even at this condition, the loading was barely sufficient to keep the sample firmly anchored on the instrument

Hardness (ASTM D-2240)

Shore D hardness was measured on compositions according to ASTM D-2240.

Glass Transition Temperature

This property was measured using a Perkin-Elmer Differential Scanning Calorimeter.

### Stress Relaxation

A standard dumbbell-shaped specimen was placed into the grips of a floor model Instron tester. It was extended at 20 in./min to a stress value of approximately 2,500 psi. The crosshead was then stopped with the chart still running. The sample was held in the stressed state from 1 to 4 hours during which time the stress decay was measured. The sample ends were sandwiched between pieces of emory cloth to prevent jaw slippage. The crosshead travel necessary to produce the 2,500 psi stress was 0.2 inch and corresponds to a specimen elongation of roughly 5-10%.

# Coefficient of Friction

Friction measurements were made on a Three Button
Sliding Contact Machine. The machine consists of an array
of three cylindrical buttons arranged in a tripod holder so
that their flat faces contact the flat face of rotating disk.
The buttons are mounted in a self-aligning fixture so that
all buttons contact the disk fully at all times. The rotating
disk is mounted on a vertical shaft, the shaft being positioned
by a slip fit through rolling contact guide bearings. Load
is applied to the end of the shaft through a dead weight and
lever system. The shaft is driven through a planetary gear
system and the torque measured mechanically through the drive

system. Coefficient of friction is determined from torque readings and dead weight load. Shaft speed is 600 rpm. Polymer samples were cut from sheet stock and adhered to brass buttons. The brass buttons were mounted in the tripod holder. The disk used in the experiments was silicon carbide with a ground surface finish.

Water lubrication was applied by directing a continuous spray at the sliding surfaces during a friction run.

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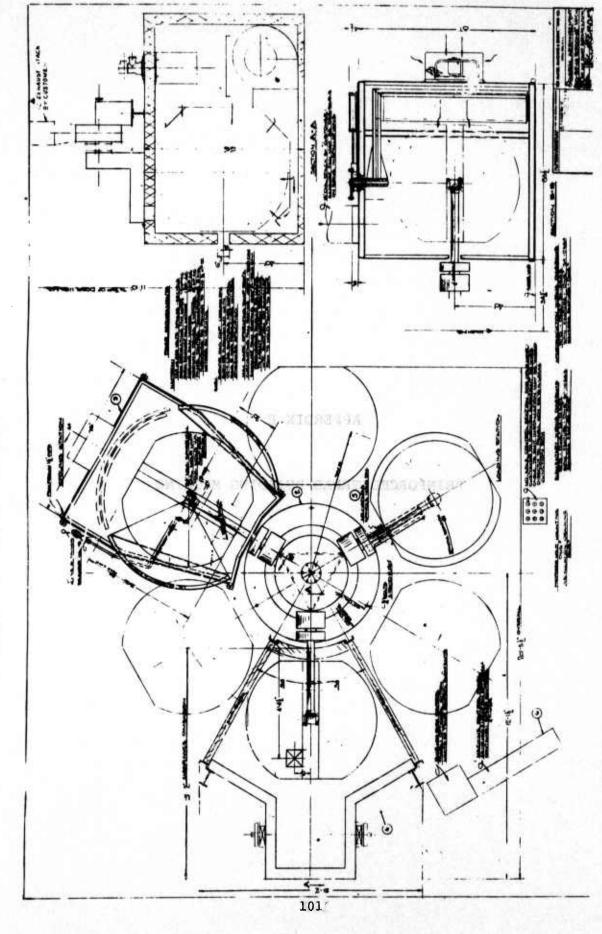
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system, configurate of friction is determined from turque credition and dead weight load. Shaft specific to fee the . The first interest and adherent in brain brains business and adherent in brains brains business with the transfer in the transfer and allow extend by load. The first used in the experiments and allows exclude with a ground sunting finish.

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APPENDIX D

ROTO CAST MACHINE

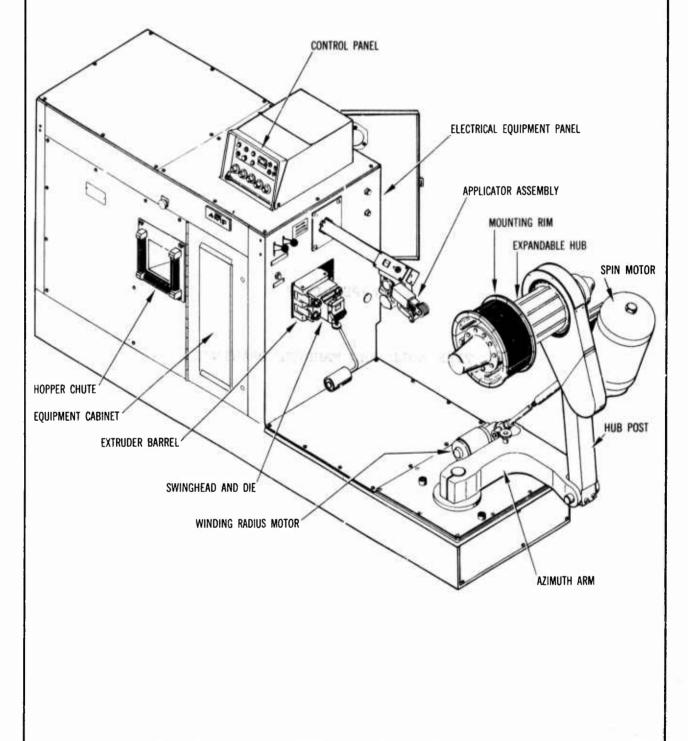


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# APPENDIX E

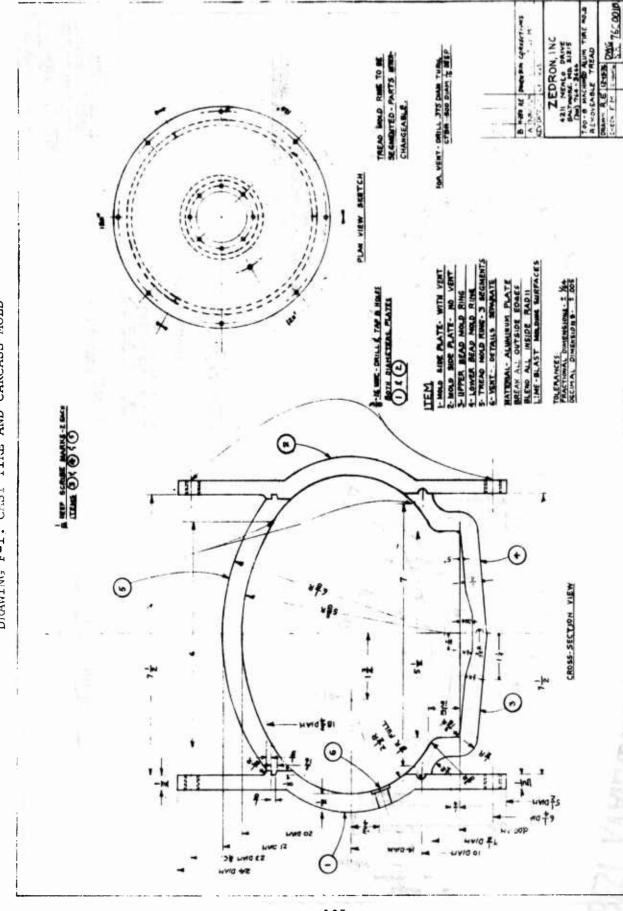
REINFORCED TREAD BUILDING MACHINE

DRAWING E-1. REINFORCED TREAD BUILDING MACHINE

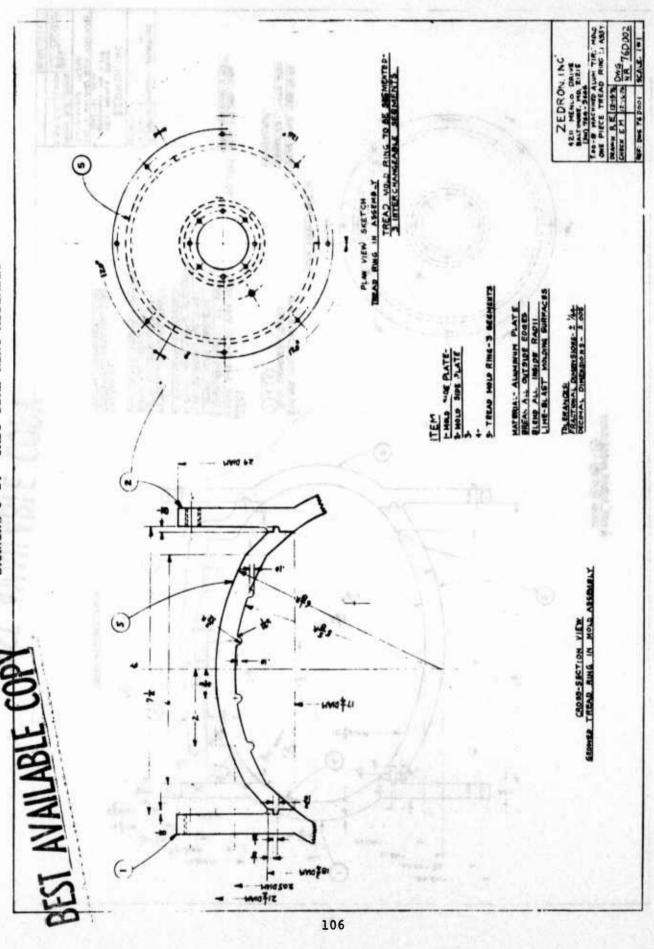


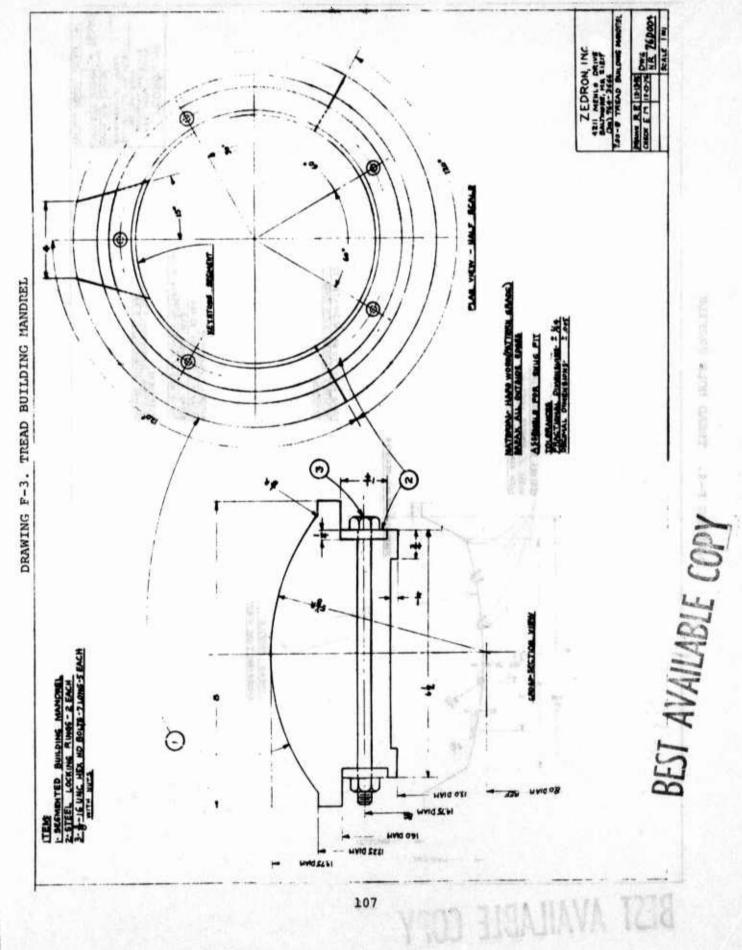
APPENDIX F

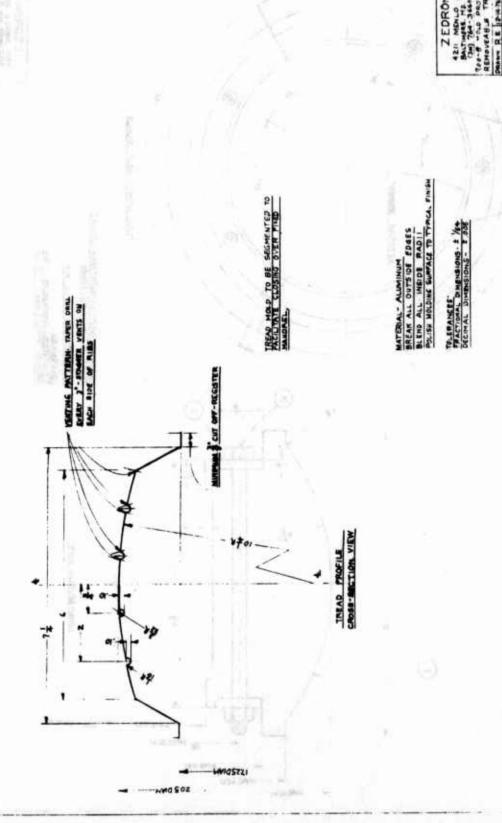
TIRE MOLD AND MANDREL DRAWINGS



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APPENDIX G

TREAD MOLD DRAWINGS

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DRAWING G-1. SEGMENTED TREAD MOLD

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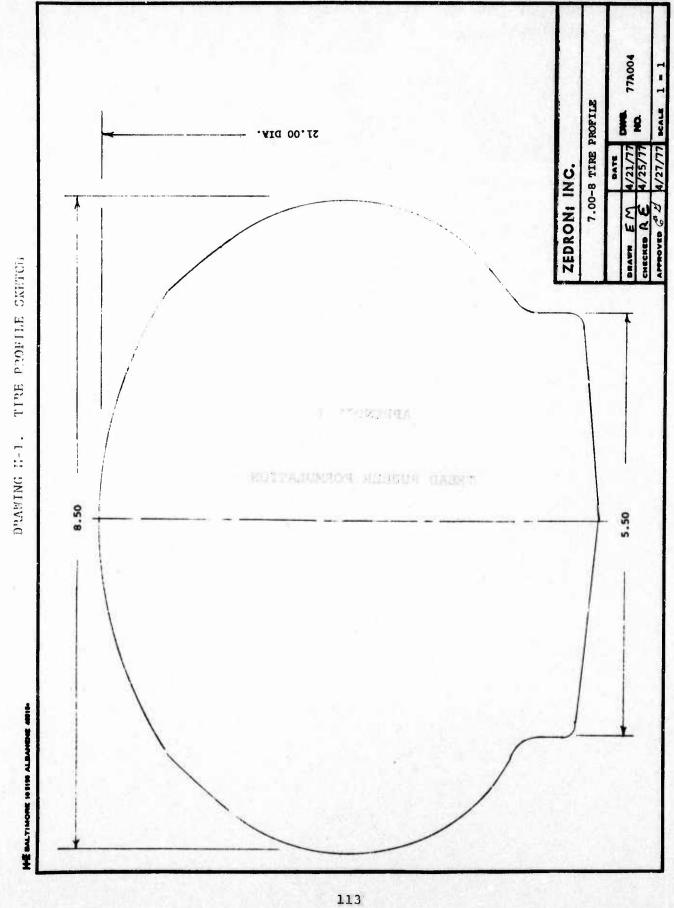
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APPENDIX H TIRE PROFILE SKETCH

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APPENDIX I

TREAD RUBBER FORMULATION

TABLE I-1 TREAD RUBBER FORMULATION

	100 RHC	% by Weight
#3 Rib Smoke Sheet	75.00	45.94
Ameripol CB or Japan Geon HX-91	25.00	15.31
R.P.A. #6 or Endor Equiv.	.07	.04
ISAF-HS (N-242)	45.00	27.56
Zinc Oxide	5.00	3.06
Stearic Acid	2.00	1.23
A Santoflex 13	1.90	.61
Santoflex AW	1.20	.73
Light Process Oil	6.00	3.68
Crystex	.50	.31
Sulfasan R	1.50	.92
Santocure MOR	1.00	.61

APPENDIX J

SIDEWALL VALVE DRAWING

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- (3) Tobolsky, A. V., <u>Properties and Structure of Polymers</u>, John Wiley and Sons, N. Y. (1960).
- (4) Guth, E., Simha, R., and Gold, O., Kolloid, Z <u>74</u>, No. 6 1936).